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REPORT OF INVESTIGATIONS—NO. 58

A STUDY OF THE EQUILIBRATION METHOD OF
DETERMINING MOISTURE IN COAL FOR
CLASSIFICATION BY RANK

BY

O. W. REES, F. H. REED, AND G. W. LAND

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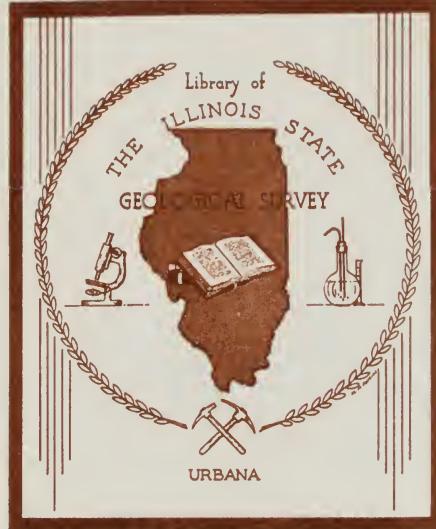


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Topographic Mapping in Cooperation with the United States Geological Survey.

This Report is a Contribution of the Section of Geochemistry, Analytical Division.



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A STUDY OF THE EQUILIBRATION METHOD OF DETERMINING MOISTURE IN COAL FOR CLASSIFICATION BY RANK

BY

O. W. REES¹, F. H. REED², and G. W. LAND³

INTRODUCTION

THE establishment, in 1936, of the tentative specifications for "Classification of Coal by Rank" (1)⁴ and the advance of these specifications to standard in 1938 (2), with the provision that the lower rank bituminous coals be classified according to their moist mineral-matter-free B.t.u. values, made imperative a reliable method for determining true bed moisture. Most of the methods employed heretofore have dealt with the empirical determination of the amount of moisture in the coal sample as presented at the laboratory without consideration of possible excess surface moisture or of moisture lost during sampling. Thus values have resulted which may or may not represent the bed moisture, which we consider to be the moisture content of the coal bed as it occurs naturally under the conditions of temperature and humidity in the mine. The equilibration procedure attempts to determine the moisture in the coal at 100 per cent humidity, which is assumed to be the true bed moisture. Such a procedure has been used in various connections but of chief interest here was its use by Lavine and others (3, 4) in studies on peat and lignite and by Stansfield and Gilbart (5) in studies on coal.

At the meeting of the Coal Classification Committee of the American Institute of Mining and Metallurgical Engineers in

New York in 1936 it was proposed that the equilibration procedure, as outlined by Stansfield and his coworkers, be made the standard procedure for determining moisture in coals having visible surface moisture for the classification of North American coals by rank. It was the opinion, however, that the reliability and convenience of the method should be further verified before it be accepted as standard. Because adoption of such a procedure as standard might invalidate moisture values obtained in the past by other methods if the values were greatly different, it therefore seemed desirable to secure information concerning the reliability of values obtained by the equilibration procedure when applied to high-moisture coals, such as are common in Illinois, and the relationship of equilibration values to values obtained by the present standard procedure. This work was undertaken to provide such information.

In this paper are presented the results obtained by applying the equilibration procedure to 21 Illinois coals ranging in moisture content from 4 to 18 per cent, as well as values obtained by other procedures, with which comparisons are made.

A brief review of the development of the equilibration procedure first proposed by Stansfield and Gilbart in 1932 (5) follows.

Dissatisfied with the usual A.S.T.M. method for air-drying coal they attempted to devise a test which would serve the dual purpose of preparing the sample for laboratory handling and for evaluating the moisture-holding capacity of the coal.

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⁴Numbers in parentheses refer to bibliography at end of report.

Their first procedure, developed in 1907 (6), required the exposure of crushed coal in shallow trays in a box which also held trays containing a solution of calcium chloride of 1.30 sp. gr. At ordinary temperatures this solution has a vapor pressure about 60 per cent that of water at the same temperature. The coal was weighed from time to time until a minimum weight was recorded. As this was a prolonged procedure, requiring six weeks in extreme cases, the method was abandoned in 1910. The second method, developed in 1923 (7), used an air-drying apparatus which Stansfield and Gilbart constructed in their laboratory. Crushed coal was dried in a rapid stream of air of 60 per cent humidity. The humidity was controlled by causing the circulating air to pass up a tube in which calcium chloride solution ran down a number of lamp wicks. By this method 48 hours were sufficient for the attainment of practical equilibrium. This apparatus was improved from time to time until 1930, but meanwhile it was found that temperature control was essential if the accuracy required for classification purposes was to be attained. Therefore, early in 1931 a new apparatus was constructed (8), in which a uniform temperature of 30° C. could be maintained and natural gas of 60 per cent humidity could be circulated. Equilibrium was reached in this new equipment within 48 hours.

The moisture value obtained after air-drying at 60 per cent humidity by the method developed in 1923 was adopted for coal classification by the Canadian Department of Customs and Excise (9). In 1931 Stansfield and Gilbart developed a vacuum-desiccator procedure for drying at constant temperature at various definite humidities (5). Their procedure consisted in equilibrating different portions of coal at different relative humidities in desiccators at a temperature of 30° C. Various saturated salt solutions were used for the corresponding relative humidities as shown in appendix A, table 1.

Residual moisture in the equilibrated samples was determined by heating at 105° C. for 3 hours in a vacuum oven in which an inert atmosphere of natural gas was maintained at an absolute pres-

sure of about 3 inches of mercury. These moisture values so obtained were plotted against relative humidity values and the curves were extrapolated to cut the 100 per cent humidity axis. The moisture values corresponding to 100 per cent humidity were taken as the "true" or "capacity" moisture values of the coal. Some work was done in which these authors attempted to obtain a satisfactory moisture value by equilibrating at only one relative humidity. In the earlier work 60 per cent humidity was used but erratic results were obtained so that in later work a relative humidity of 97 per cent was used. Moisture values obtained at a single humidity level were used only after one or more entire moisture humidity curves for the various coal areas in the province had been obtained. A study of 54 entire humidity curves of coals with moisture content ranging from 1 to 32 per cent showed that the moisture retained at 97 per cent humidity averaged 98.6 per cent of the extrapolated "true" moisture value and seldom varied far from this value. They therefore calculated "true" moisture by dividing the moisture retained at 97 per cent humidity by 0.986 in all cases where they did not prepare the entire curve. These values were termed moisture "by calculation." In some cases the moisture humidity curves could be extrapolated easily whereas in other cases irregular curves were obtained, the extrapolation of which was impossible. A distinct difference between dehydration and rehydration curves was found.

ACKNOWLEDGMENTS

The writers wish to acknowledge the cooperation and assistance of the management of the mines in collecting samples. To Dr. G. H. Cady they express their appreciation for many helpful suggestions during the preparation of this report. The help of Dr. L. C. McCabe and C. C. Boley in securing samples, and of J. W. Robinson, P. E. Grotts and M. L. Kalinowski in securing analytical data is gratefully acknowledged. To Dr. M. M. Leighton, Chief of the Survey, is due the credit for making this investigation possible.

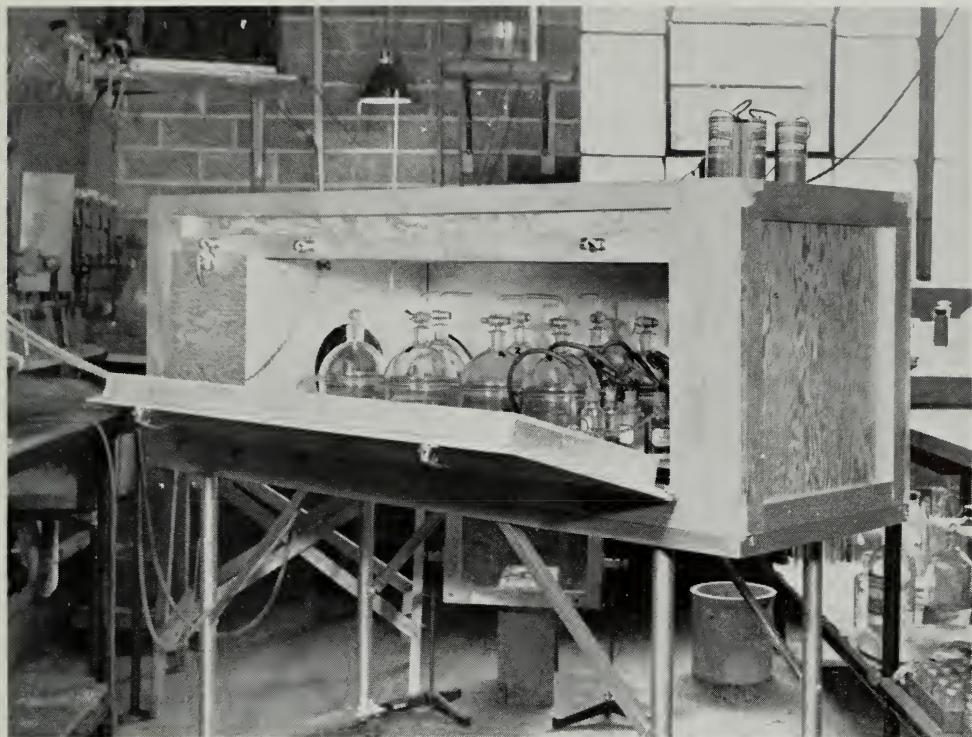


FIG. 1.—Air-bath thermostat.

EXPERIMENTAL WORK

The experimental work of this report was an application of the equilibration method of Stansfield and Gilbart for the determination of bed moisture of Illinois coal. The procedure adopted and the equipment used for the first six coals studied were as nearly as possible those outlined by Stansfield and Gilbart. Moisture determinations for these six samples of coal were made, using a large air oven to provide constant temperature during equilibration. Desiccators were evacuated every twelve hours. The discrepancies in the results obtained indicated inadequate temperature control. Therefore, a constant-temperature water-bath was substituted for this air oven and equilibrations on 15 other coal samples were made. Again, desiccators were evacuated every twelve hours. In addition, equilibrations were made at three or four humidities on these 15 samples using a nitrogen atmosphere without evacuation. Below is a description of the apparatus and an outline of the procedure followed in the work.

APPARATUS

A double-walled box approximately 72 by 26 by 24 inches, outside dimensions, was built to serve as a constant temperature oven for the equilibrations. The walls of this box were insulated with about three inches of rock wool, heat was provided by light bulbs so placed that air from a fan was heated at one end of the box, passed between the upper walls to the other end and was admitted to the chamber proper through a plate in which many holes were drilled to give good distribution. A cooling coil, through which cool water was passed continuously, was provided so that the heating bulbs worked against this cooling arrangement. A mercury thermo-regulator was used to control the temperature of the box at $30^{\circ}\text{ C.} \pm 0.5^{\circ}\text{ C.}$ The box (fig. 1) was equipped with a side door over its entire length for convenience in putting in and taking out desiccators. Later equilibrations were made in a large water-bath thermostat (fig. 2) at $30^{\circ}\text{ C.} \pm 0.1^{\circ}\text{ C.}$

Pyrex vacuum desiccators as shown in the oven (fig. 1) were used for the equili-

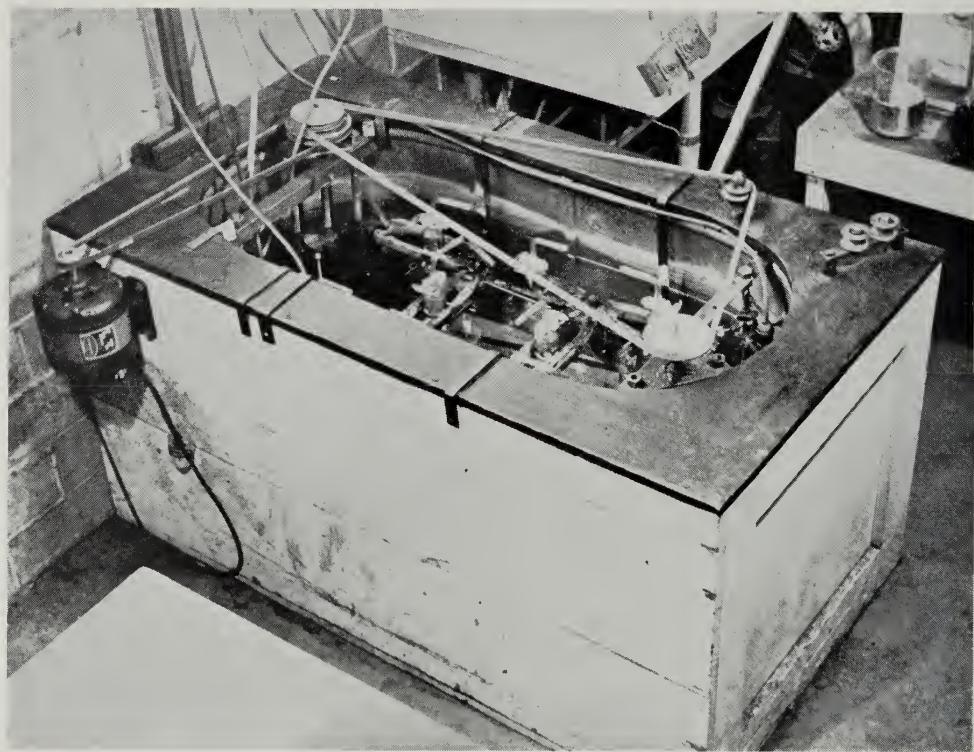


FIG. 2.—Water-bath thermostat.

brations. For determining moisture after equilibration a Cenco vacuum oven was used.

Small petri dishes were used to hold the samples during equilibration. Lids were provided for these dishes to protect the samples during weighing and transfer from one container to another. Bottles containing saturated solutions of the same salts as those used in the desiccators were used to provide properly humidified air to desiccators in bringing the pressure to atmospheric pressure at the end of the equilibration periods.

PROCEDURE

In studying the application of the Stansfield and Gilbart method to the determination of moisture in Illinois coals, it was decided to use fresh face-samples of low, medium, and high moisture content. Accordingly, six samples of coal were collected at intervals of about one week and each treated as follows:

1) A sample was cut down from the face, crushed and ground to $\frac{1}{4}$ inch size,

riffled to one quart in the mine and brought to the laboratory where it was further crushed to minus 14-mesh, and portions were weighed and equilibrated for 48 hours at each of the nine different humidities recommended by Stansfield and Gilbart.

After equilibration, the samples were reweighed and then dried at 105° C. in the vacuum oven for three hours. The pressure in the oven was maintained at about three inches of mercury by admitting a slow stream of nitrogen. At the end of the three-hour period the samples were removed from the oven, cooled, weighed, and the moisture values calculated. The samples of coal were then re-equilibrated for 48 hours at the same humidities after which moisture values were determined again. Both first and second equilibration moisture-values were plotted against humidity, the curves were extrapolated to cut the 100 per cent humidity axis, and the moisture value obtained was taken as the "true" moisture value of the sample.

2) Another sample was collected exactly the same as that for 1) for a proximate analysis. On this sample air dry loss and the regular A.S.T.M. (10) moisture were determined to give total moisture.

3) A 50 to 75 lb. gross sample was taken from the freshly exposed face and brought to the laboratory in a sealed container, where it was crushed to $\frac{1}{4}$ inch size and rifled down to a quart sample. Moisture was then determined by the usual A.S.T.M. procedure including air dry loss, etc.

4) Two 20-mesh samples (approximately 5 grams each) were prepared in the mine and placed in weighing bottles which had been previously weighed in the laboratory. These samples were brought to the laboratory where they were weighed and *total moisture* values were determined by the A.S.T.M. procedure (10).

5) Two 20-mesh samples were prepared in the mine exactly as in 4) and these samples were used for total moisture determinations in the vacuum oven following the same procedure used for determining moisture in the equilibrated samples.

In all cases except 3) duplicate portions were taken as a precaution and duplicate determinations were made in all cases. Later, fifteen additional coal samples were obtained and treated by the same procedure as outlined above, with the exception that a water-bath thermostat was used for the equilibrations in place of the air-bath thermostat. In addition, portions of these samples were equilibrated at three or four humidities in a nitrogen atmosphere without evacuation in the air-bath thermostat.

RESULTS

Equilibration results for the first six coals studied are given in table 1 and shown graphically in fig. 3. Table 2 presents proximate analyses of these coals together with a comparison of moisture results obtained by different procedures. Tables 3, 4, 6, and 7 and figures 4, 5, and 6 present data for the 15 samples equilibrated in the water thermostat. Tables 3, 5, 6, and 8 and figures 7, 8, and 9 present data for the 15 samples equilibrated in nitrogen atmosphere, with no evacuation, in the air thermostat.

DISCUSSION OF RESULTS

Six coals were first equilibrated at nine different humidities, and the moisture content of each sample was determined in the vacuum oven. These values are listed in table 1 as "Residual moisture in per cent after first equilibration." The dry samples were then re-equilibrated and moisture values were determined again. These are listed in table 1 as "Residual moisture in per cent after second equilibration." A comparison of equilibration values shows that they are in close agreement in the low humidities, diverge considerably at the intermediate humidities, and converge again at the high humidities. The values obtained for the second equilibration are, in general, lower than those of the first equilibration, the variation being greatest for the intermediate humidities.

Extrapolation of the moisture-humidity curves to give 100 per cent humidity values gave results of questionable validity. In the case of sample C-1901A (fig. 3), the slope of the curve at the high humidities was so steep that extrapolation gave a result which appeared questionable. In the case of sample C-1864A (fig. 3), strict adherence to all values gave a curve proceeding downward at the point of crossing the 100 per cent humidity axis. While the moisture value for the sample equilibrated at 97.7 per cent humidity was lower than that for the 96.8 per cent humidity by an amount within experimental error, it leaves one in doubt as to which way to extend the curve to cut the 100 per cent humidity axis. Data obtained on the second equilibration series on samples C-1901A and C-1904A (fig. 3) gave such steep curves at the higher humidities that values obtained by their extrapolation seem questionable.

Attempts to draw smooth curves through the points as plotted were unsuccessful, as wavy curves resulted which were not easily extrapolated. Therefore, all points were connected by straight lines. The slope of the curve as extrapolated to 100 per cent humidity was influenced little if any by the low or intermediate humidity values, but was determined by the last two high humidity moisture values in most cases.

EQUILIBRATION METHOD OF

TABLE 1.—EQUILIBRATION DATA FOR FIRST SIX SAMPLES STUDIED

Lab. No.	County	Coal bed No.	Equili- bra- tion ¹	HUMIDITY VALUES (PER CENT)							CALCULATED MOISTURE						
				11	20	46.8	59.7	75.1	84.5	89.9	96.8	97.7	100 ²	96.8% 100% ³	97.7% 100% ³		
C-1863A	LaSalle	2	A	1.4	2.0	4.4	6.4	11.8	13.0	13.6	14.3	14.3	14.3	1.000	1.000	14.7	
C-1864A	Perry	6	A	1.2	2.0	2.6	3.1	4.8	7.8	11.9	13.8	14.0	14.4	.958	.972	15.4	
C-1900A	Saline	5	A	1.9	1.5	2.4	3.4	3.9	6.0	8.3	9.6	10.0	10.0	1.000	.990	10.7	
C-1901A ⁵	Henry	6	A	1.2	1.7	2.4	2.6	3.2	4.5	5.5	6.1	6.7	6.7	1.000	.990	10.5	
C-1903A	St. Clair	6	A	1.7	2.2	2.4	3.3	7.5	14.3	15.9	16.3	16.6	17.1	18.1	.917	.945	17.1
C-1904A ⁵	Henry	6	A	1.4	2.1	2.9	3.2	4.8	6.8	8.7	9.0	10.0	10.4	.942	.962	10.1	
Average	Average	...	A	1.4	2.4	4.6	7.3	14.4	16.4	17.3	18.0	18.2	18.6	.840	.978	10.8	
		...	B	1.4	2.6	3.3	3.8	6.0	9.7	13.0	17.0	17.2	18.8	.904	.915	18.5	
		...	A971	.979	18.9		
		...	B898	.930		

¹A Residual moisture in per cent after first equilibration.²B Residual moisture in per cent after second equilibration.³By extrapolation.⁴Factors 0.971 and 0.898 used respectively for 1st and 2nd equilibration values.⁵Factors 0.979 and 0.930 used respectively for 1st and 2nd equilibration values.

Strip mine.

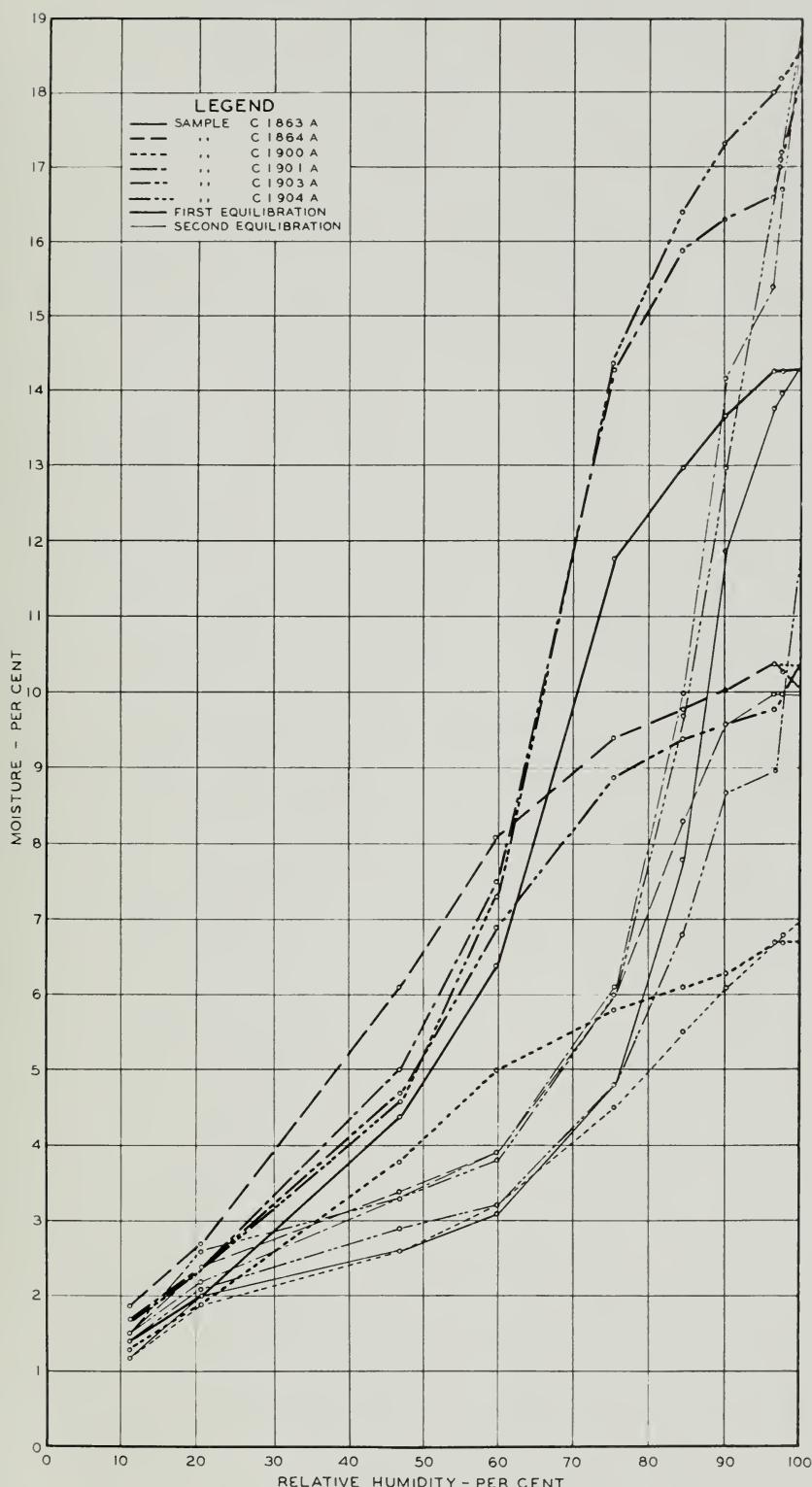


Fig. 3.—Moisture-humidity curves for first six coals studied.

EQUILIBRATION METHOD OF

TABLE 2.—ANALYSES AND MOISTURE VALUES OBTAINED BY DIFFERENT PROCEDURES
FOR FIRST SIX SAMPLES STUDIED

Lab. No.	County	Coal bed No.	As _{SH}		Volatile		Fixed Carbon		Sulfur		B.T.U.		Moisture Values Obtained by Different Methods ¹							
			As rec'd	Dry	As rec'd	Dry	As rec'd	Dry	As rec'd	Dry	Unit coal	A	B	C	D	E	F			
C-1863A	LaSalle	...	2	10.1	11.8	37.2	43.5	38.1	44.7	3.9	4.5	10.791	12.630	14.6	14.2	14.4	14.7	14.3	14.4	
C-1864A	Perry	...	6	10.8	12.0	36.0	40.2	42.7	47.8	3.9	4.4	11.010	12.304	14.279	10.5	9.9	10.2	10.5	10.4	10.0
C-1900A ²	Saline	...	5	8.4	9.0	34.3	36.7	50.7	54.3	2.5	2.7	12.410	13.290	14.810	7.2	6.6	7.1	7.2	6.7	7.0
C-1901A ³	Henry	...	6	9.8	11.9	34.5	41.9	38.0	46.2	3.8	4.7	9.964	12.113	14.045	18.4	17.7	18.7	19.1	18.1	19.0
C-1903A ²	St. Clair	...	6	11.0	12.4	38.1	43.0	39.6	44.6	3.7	4.2	11.000	12.400	14.460	10.5	10.6	10.4	10.8	10.4	11.9
C-1904A ³	Henry	...	6	10.0	12.6	32.1	40.5	37.2	46.9	3.3	4.1	9.635	12.151	14.200	20.7	19.5	20.4	20.8	18.6	18.8

¹A Sample ground to $\frac{1}{4}$ -inch in mine. Total moisture by Air Dry Loss + Regular Moisture.

B 50-lb. mine sample crushed to $\frac{1}{4}$ -inch in laboratory. Total moisture by Air Dry Loss + Regular Moisture.

C 5-gram sample crushed to 20 mesh in mine. Moisture by A.S.T.M. oven, air atmosphere, 105° C., $1\frac{1}{2}$ hrs.

D 5-gram sample crushed to 20 mesh in mine. Moisture by vacuum oven, 3 inches Hg., N_2 atmosphere, 105° C., 3 hours.

E 100 per cent humidity extrapolated moisture values, 1st equilibration.

F 100 per cent humidity extrapolated moisture values, 2nd equilibration.

²County average values from Illinois State Geological Survey Bulletin No. 62. No proximate analyses of individual samples made at strip mine.

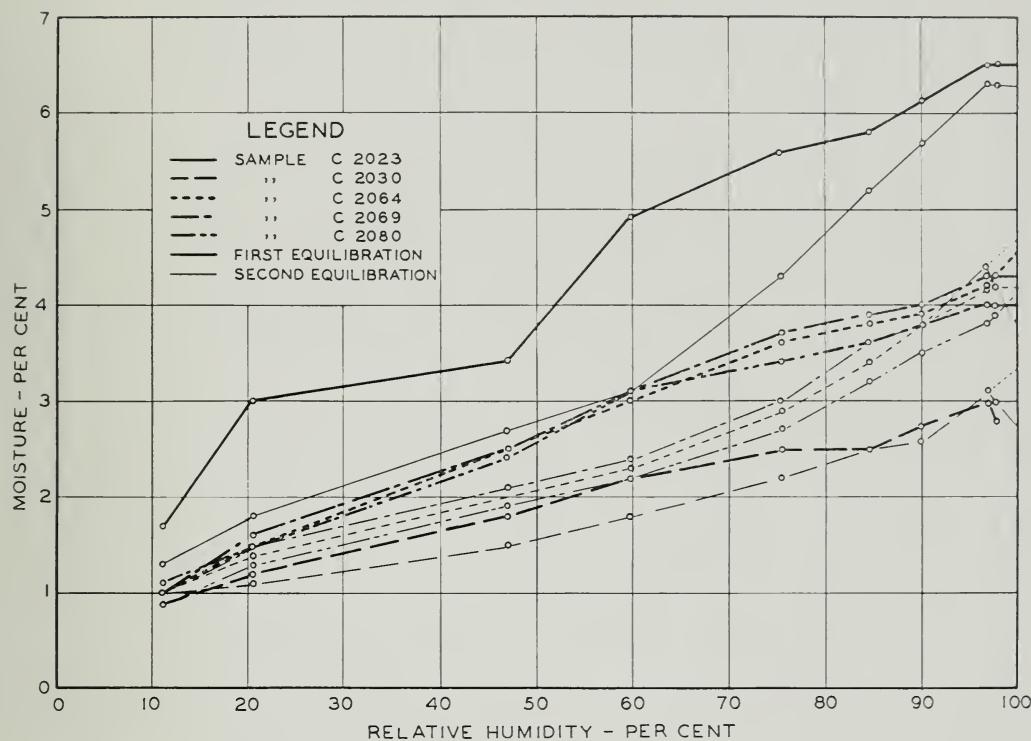


FIG. 4.—Moisture-humidity curves for low-moisture samples, desiccators evacuated, water-bath thermostat.

TABLE 3.—ANALYSES OF FIFTEEN SAMPLES USED FOR FURTHER EQUILIBRATION STUDIES

Lab. No.	County	Coal bed No.	ASH		VOLATILE		FIXED CARBON		SULFUR		B.T.U.	
			As rec'd	Dry	As rec'd	Dry	As rec'd	Dry	As rec'd	Dry	As rec'd	Dry
C-2009	Vermilion.....	7	10.1	11.7	35.9	41.4	40.6	46.9	3.10	3.58	10.981	12.675
C-2010	La Salle.....	2	9.0	10.5	37.0	43.2	39.8	46.3	3.04	3.54	11.085	12.920
C-2022	Logan.....	5	11.4	13.3	34.0	39.7	40.2	47.0	2.84	3.32	10.571	12.344
C-2023	Saline.....	5	9.3	10.1	32.8	35.5	50.2	54.4	2.98	3.22	12.106	13.113
C-2030	Gallatin.....	5	11.1	11.5	36.7	38.2	48.4	50.3	3.39	3.52	12.640	13.136
C-2031	St. Clair.....	6	11.6	13.0	37.0	41.5	40.5	45.5	3.69	4.14	11.005	12.355
C-2034	Franklin.....	6	8.4	9.3	33.1	36.6	49.0	54.1	1.16	1.28	11.804	13.038
C-2037	Christian.....	6	11.2	12.9	35.9	41.3	39.9	45.8	4.48	5.15	10.624	12.205
C-2039	Marion.....	6	9.8	11.3	34.9	39.9	42.8	48.8	3.31	3.79	11.000	12.572
C-2042	Randolph.....	6	10.7	12.0	35.1	39.4	43.3	48.6	2.80	3.14	11.101	12.462
C-2046 ¹	Knox.....	6	9.1	11.1	32.2	39.2	40.7	49.7	2.95	3.60	10.248	12.492
C-2059 ¹	La Salle.....	(2)	6.5	7.7	35.1	41.5	43.1	50.8	2.15	2.54	11.355	13.411
C-2064	Gallatin.....	5	9.0	9.4	36.7	38.4	49.8	52.2	3.19	3.34	12.736	13.330
C-2069	Saline.....	5	11.5	12.1	35.0	36.7	48.8	51.2	3.30	3.46	12.248	12.846
C-2080	Saline.....	5	12.3	12.9	36.2	38.0	46.7	49.1	3.49	3.67	12.101	12.706

¹Strip mine.

²Local below coal No. 6.

EQUILIBRATION METHOD OF

TABLE 4.—DATA ON SAMPLES EQUILIBRATED IN EVACUATED DESICCATORS IN CONSTANT TEMPERATURE (30° C.) WATER BATH

Lab. No.	County	Coal bed No.	Equili- bra- tion ¹	HUMIDITY VALUE (PER CENT)								96.7% 100% ²	97.7% 100% ²	CALCULATED MOISTURE Value 1 ³	CALCULATED MOISTURE Value 2 ⁴
				11.2	20.6	46.9	59.8	75.4	84.4	90.1	96.7	97.7			
C-2009...	Vermilion...	7	A B	1.8 1.4	2.0 2.8	4.0 3.3	6.8 5.1	10.0 7.5	11.5 11.1	12.4 14.0	12.9 14.0	13.1 13.7	12.7 12.7	13.1 13.3	13.4 13.3
C-2010...	La Salle...	2	A B	1.6 1.4	2.1 1.9	3.6 2.7	5.1 5.6	10.4 8.0	11.4 12.0	13.1 13.1	14.0 14.4	14.0 14.7	14.0 14.7	14.0 14.7	14.6 14.6
C-2022...	Logan...	5	A B	1.7 1.5	2.5 2.0	3.4 3.4	4.3 4.9	7.0 5.6	10.7 6.1	12.9 5.8	13.8 14.2	13.9 14.4	14.1 14.9	14.5 15.2	14.5 15.3
C-2023...	Saline...	5	A B	1.7 1.3	3.0 1.8	3.4 2.7	4.9 4.3	7.0 5.2	10.7 5.7	12.9 6.3	13.7 6.3	13.9 6.3	13.9 6.3	14.9 14.9	15.0 15.0
C-2030...	Gallatin...	5	A B	1.9 1.0	1.2 1.5	1.8 1.8	2.2 2.2	2.5 2.5	2.5 2.5	2.8 3.0	3.0 3.1	3.0 3.0	3.2 3.3	3.2 3.3	3.2 3.1
C-2031...	St. Clair...	6	A B	1.9 1.3	2.3 1.9	4.1 3.0	6.6 3.4	8.5 5.0	8.5 7.1	9.6 9.1	10.1 10.1	9.7 10.4	10.4 10.4	10.6 10.6	10.6 10.6
C-2034...	Franklin...	6	A B	2.0 3.8	2.7 2.4	4.5 3.7	7.6 6.7	8.4 8.4	9.0 9.1	9.3 9.7	9.8 9.7	9.8 9.7	9.8 9.7	10.0 10.0	10.2 10.2
C-2037...	Christian...	6	A B	2.2 1.6	2.2 2.0	4.6 3.8	7.9 7.4	10.0 7.4	11.4 10.1	11.8 11.6	12.4 12.8	13.0 12.7	14.3 13.4	13.1 13.4	13.5 13.3
C-2039...	Marion...	6	A B	2.0 1.7	2.2 2.2	5.4 4.5	7.5 6.7	9.5 8.4	11.2 10.2	11.5 11.2	12.8 13.4	13.8 14.0	14.0 14.5	14.4 14.6	14.4 14.6
C-2042...	Randolph...	6	A B	2.0 1.5	2.6 2.0	5.3 3.4	8.0 3.9	9.5 6.0	10.0 8.3	10.3 9.4	10.9 10.3	10.8 10.6	10.9 11.1	10.0 10.8	10.2 11.1
C-2046 ⁵ ...	Knox...	6	A B	2.0 1.7	2.6 2.2	5.8 3.7	8.7 6.9	15.3 10.0	16.6 12.7	17.4 17.7	18.0 18.0	18.6 18.6	19.3 19.3	18.6 18.6	18.8 18.8
C-2059 ⁵ ...	La Salle...	(6)	A B	1.7 1.4	2.2 1.9	4.5 3.0	7.1 3.4	11.3 5.1	13.0 7.7	13.4 9.7	14.4 14.1	14.6 14.2	14.9 14.4	14.6 14.6	14.6 14.6
C-2064...	Gallatin...	5	A B	1.0 1.0	1.5 1.0	2.5 1.4	3.0 2.3	3.0 2.9	3.6 3.4	3.9 3.8	4.2 4.2	4.5 4.2	4.5 4.2	4.4 4.4	4.4 4.4
C-2069...	Saline...	5	A B	1.0 1.0	1.6 1.5	2.5 2.1	3.1 2.4	3.7 3.0	3.9 3.6	4.0 3.8	4.3 4.2	4.3 4.2	4.3 4.2	4.5 4.4	4.5 4.4
C-2080...	Saline...	5	A B	1.1 .9	1.5 1.3	2.4 1.9	3.1 2.2	3.4 2.7	3.6 3.2	3.8 3.5	4.0 3.9	4.0 4.1	4.0 3.9	4.6 4.6	4.2 4.2
Average...	Average...	...	A B

¹A Residual moisture in per cent after 1st equilibration.²B Residual moisture in per cent after 2nd equilibration.³By extrapolation.⁴Factors 0.960 and 0.957 used respectively for 1st and 2nd equilibration values.⁵Local below coal No. 6.

TABLE 5.—DATA ON SAMPLES EQUILIBRATED IN ATMOSPHERE OF NITROGEN AT ATMOSPHERIC PRESSURE IN CONSTANT TEMPERATURE (30° C.) AIR BATH

Lab. No.	County	Coal bed No.	Equili- bration (¹)	HUMIDITY VALUES (PER CENT)					Ratio of moisture values 97.7% 100%	Calcu- lated moisture value ³
				11.2	59.8	84.4	97.7	100 ²		
C-2009	Vermilion.....	7	A	1.6	7.1	11.2	13.0	13.3	.977	13.3
			B	1.4	3.4	7.2	12.5	13.3	.940	13.0
C-2010	La Salle.....	2	A	1.6	13.2	14.2	14.4	.986	14.5
			B	1.4	7.8	13.4	14.3	.937	14.0
C-2022	Logan.....	5	A	1.7	13.4	14.4	14.6	.986	14.7
			B	1.5	9.6	14.1	14.8	.953	14.7
C-2023	Saline.....	5	A	1.6	6.0	6.5	6.6	.985	6.6
			B	1.3	5.1	6.2	6.4	.969	6.5
C-2030	Gallatin.....	5	A	.9	2.2	2.7	3.0	3.1	.968	3.1
			B	.8	1.7	2.4	3.0	3.1	.968	3.1
C-2031	St. Clair.....	6	A	1.9	9.4	10.7	10.9	.982	10.9
			B	1.3	6.8	9.9	10.4	.952	10.3
C-2034	Franklin.....	6	A	2.0	7.9	9.0	10.1	10.3	.981	10.3
			B	1.7	4.7	8.4	9.9	10.1	.980	10.3
C-2037	Christian.....	6	A	1.9	8.8	11.6	13.2	13.5	.978	13.5
			B	1.6	4.9	9.6	12.7	13.2	.962	13.2
C-2039	Marion.....	6	A	2.0	9.4	11.4	13.7	14.1	.972	14.0
			B	1.6	5.2	10.1	12.9	13.4	.963	13.5
C-2042	Randolph.....	6	A	1.9	8.4	10.0	10.8	10.9	.991	11.0
			B	1.5	4.1	8.3	10.5	10.9	.963	10.9
C-2046 ⁴	Knox.....	6	A	1.9	9.2	16.4	17.6	17.8	.989	17.9
			B	1.7	4.2	9.5	16.5	17.6	.938	17.2
C-2059 ⁴	La Salle.....	(5)	A	1.7	7.8	13.3	14.5	14.7	.986	14.8
			B	1.4	3.4	7.1	13.3	14.3	.930	13.9
C-2064	Gallatin.....	5	A	1.1	3.2	3.8	4.2	4.3	.977	4.3
			B	1.1	2.4	3.3	4.1	4.2	.976	4.3
C-2069	Saline.....	5	A	1.1	3.2	3.8	4.2	4.3	.977	4.3
			B	1.0	2.3	3.3	4.1	4.2	.976	4.3
C-2080	Saline.....	5	A	1.0	3.0	3.5	3.9	4.0	.975	4.0
			B	.9	2.1	3.0	3.7	3.8	.974	3.9
Average			A981	
Average			B959	

¹A Residual moisture after 1st equilibration.²B Residual moisture after 2nd equilibration.³By extrapolation.⁴Factors 0.981 and 0.959 used respectively for 1st and 2nd equilibration values.⁵Strip mine.⁶Local below coal No. 6.

In table 2 are tabulated moisture values for these six samples obtained by six procedures including first and second equilibrations on each sample. These values, in general, check very well with the exception that the second equilibration value for sample C-1903A is higher than the other values and both the first and second equilibration values for sample C-1904A are lower than the other values. The first and second equilibration values check reasonably well in four of the six samples but for samples C-1901A and C-1903A there is considerable variation.

The difficulties which arose in applying the equilibration procedure and interpreting the results on the first six coals led us to study the vapor pressures of the solutions in desiccators used for equilibration. Vapor pressures were determined for each solution as used in the desiccator and then relative humidity values were calculated. A description of apparatus and procedure used in these determinations is presented in appendix A. Information was obtained in the course of these determinations which has a distinct bearing on the equilibration procedure.

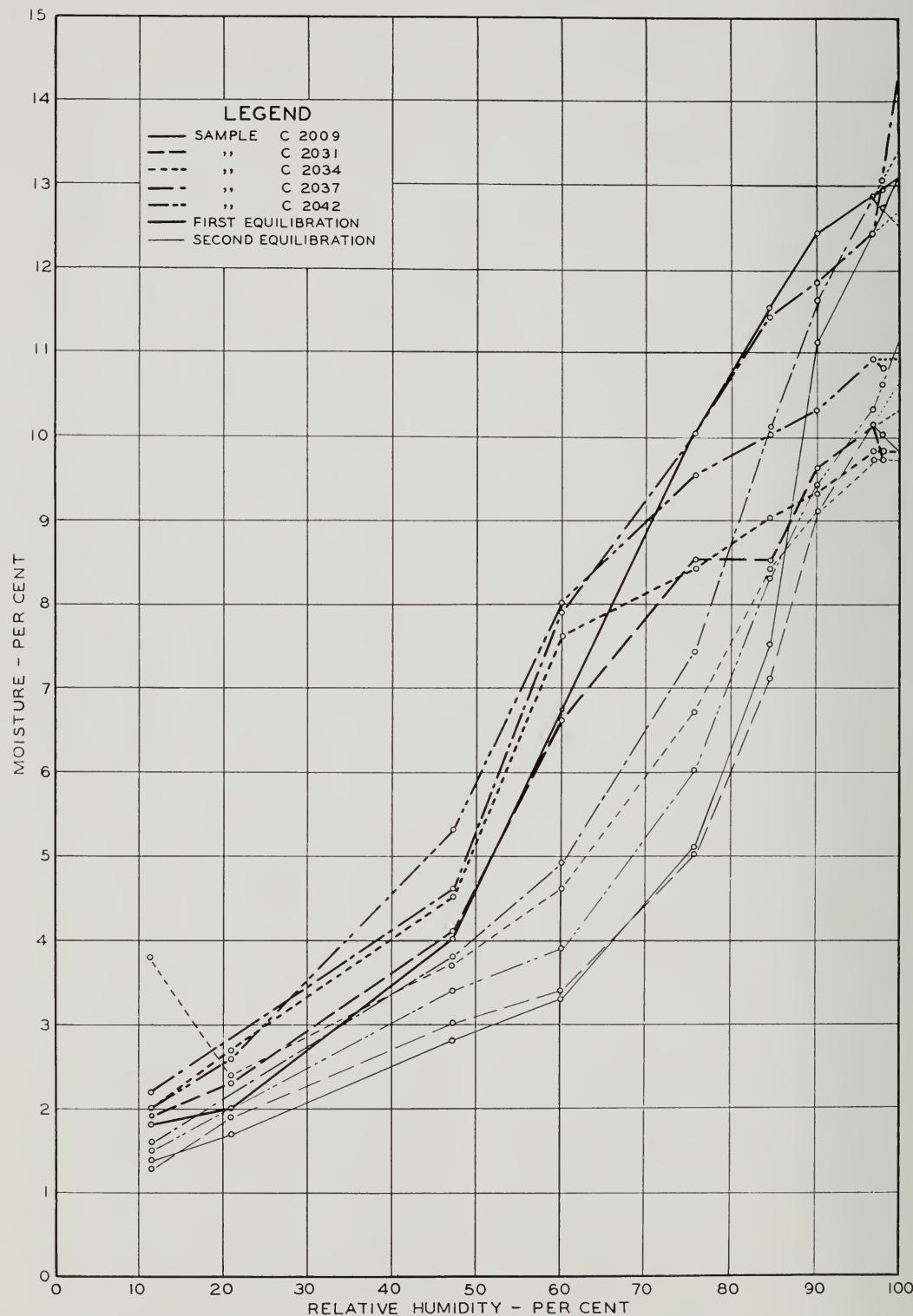


FIG. 5.—Moisture-humidity curves for medium-moisture samples, desiccators evacuated, water-bath thermostat.

TABLE 6.—MOISTURE VALUES OBTAINED BY DIFFERENT METHODS FOR FIFTEEN SAMPLES

Lab. No.	County	Coal bed No.	MOISTURE VALUES OBTAINED BY DIFFERENT METHODS ¹ (per cent)								Mine humidity (per cent) ²
			A	B	C	D	E	F	G	H	
C-2009	Vermilion....	7	13.3	12.6	13.5	14.0	13.1	13.1	13.3	13.3	95
C-2010	La Salle....	2	14.3	13.1	14.4	14.9	14.0	14.1	14.4	14.3	96
C-2022	Logan....	5	14.4	13.8	14.6	15.1	15.2	14.9	14.6	14.8	94
C-2023	Saline....	5	7.7	7.7	7.3	8.1	6.5	6.3	6.6	6.4	97
C-2030	Gallatin....	5	3.8	3.7	3.1	3.7	3.1	3.3	3.1	3.1	92
C-2031	St. Clair....	6	10.9	10.4	11.5	10.4	10.6	10.9	10.4	95
C-2034	Franklin....	6	9.5	9.4	9.4	10.0	9.8	9.7	10.3	10.1	97
C-2037	Christian....	6	13.0	12.0	12.9	13.4	14.3	13.4	13.5	13.2	97
C-2039	Marion....	6	12.5	12.3	12.5 ³	13.1 ³	17.1	15.2	14.1	13.4	93
C-2042	Randolph....	6	10.9	10.2	11.1	11.6	10.9	11.1	10.9	10.9	92
C-2046 ⁴	Knox....	6	18.0	17.9	17.6	18.8	18.6	19.3	17.8	17.6	88 ⁵
C-2059 ⁴	La Salle....	(6)	15.1	15.1	14.5	15.1	14.9	14.4	14.7	14.3	48 ⁵
C-2064	Gallatin....	5	5.0	5.0	4.8	5.1	4.5	4.2	4.3	4.2	97
C-2069	Saline....	5	4.8	4.8	4.7	5.1	4.3	4.7	4.3	4.2	96
C-2080	Saline....	5	5.0	5.0	4.8	5.1	4.0	4.1	4.0	3.8	96

¹A Sample ground to $\frac{1}{4}$ -inch in mine. Total moisture by Air Dry Loss + Regular Moisture.²B 50-lb. mine sample crushed to $\frac{1}{4}$ -inch in laboratory. Total moisture by Air Dry Loss + Regular Moisture.³C 5-gram sample crushed to 20 mesh in mine. Moisture by A.S.T.M. oven, air atmosphere, 105° C. 1½ hours.⁴D 5-gram sample crushed to 20 mesh in mine. Moisture by vacuum oven 3 inches Hg., N₂ atmosphere, 105° C., 3 hours.⁵E 100 per cent humidity extrapolated moisture values—1st equilibration, evacuated.⁶F 100 per cent humidity extrapolated moisture values—2nd equilibration, evacuated.⁷G 100 per cent humidity extrapolated moisture values—1st equilibration, nitrogen atmosphere.⁸H 100 per cent humidity extrapolated moisture values—2nd equilibration, nitrogen atmosphere.²Determined with sling psychrometer.³Crushed to 20 mesh in mine; brought to laboratory in large sample bottle and transferred to weighing bottle.⁴Strip mine.⁵Outdoor humidity.⁶Local below coal No. 6.

An attempt to use the original air oven was not successful since it was impossible to hold the temperature of the desiccators sufficiently close to 30° C. by this means. It was therefore necessary to use a water-bath thermostatically controlled at 30° C. varying not more than $\pm 0.1^\circ$ C. Evacuation of the desiccators lowers the temperature 5° C. or more and in the air oven, several hours are necessary to regain the proper temperature. Such lowering of temperature with slow return to proper temperature, is important both in making vapor pressure determinations, and in the actual equilibration of coal samples. Lowering of the temperature by evacuation results in condensation of moisture on the coal sample and unless it is left until equilibrium is regained, the moisture of the sample will be too high. This is particularly important at the higher humidities. The use of a water-bath thermostat was found to effect return to the desired temperature within a short time (about one-half hour) after evacuation and proved satisfactory in the vapor pressure determinations. The humidity values calculated from the de-

termined vapor pressure values checked closely with those used by Stansfield and Gilbart. The humidity values used by them and those determined in this laboratory are compared in table I, appendix A.

Having proved to our satisfaction that the relative humidities in the desiccators used were satisfactory, provided there was adequate temperature control, additional coal samples were studied. Fifteen samples representing low, medium, and high moisture coals of the State were obtained and treated as outlined above. Analyses of these samples are shown in table 3. The samples were equilibrated for 48 hours in desiccators placed in a water-bath thermostat, with evacuation every 12 hours. Results of these determinations are tabulated in table 4 and shown graphically in figures 4, 5 and 6.

As stated above, it was learned that evacuation lowered the temperature in the desiccators and that the return to the desired temperature was slow in an air-bath thermostat. It was thought that elimination of evacuation and the use of an inert atmosphere at normal pressure

TABLE 7.—RESIDUAL MOISTURE VALUES AFTER SECOND EQUILIBRATION (DETERMINED) AND PER CENT INCREASE IN WEIGHT OF SAMPLES DURING SECOND EQUILIBRATION. WATER BATH—DESICCATORS EVACUATED

Lab. No.	County	Coal bed No.	Humidity Values (Per Cent)														
			11.2			20.6			46.9			59.8			75.4		
			Wt. ¹	R.M. ²	Wt. ¹	R.M. ²	Wt. ¹	R.M. ²	Wt. ¹	R.M. ²	Wt. ¹	R.M. ²	Wt. ¹	R.M. ²	Wt. ¹	R.M. ²	
C-2009	Vermilion	7	1.4	1.4	1.7	1.7	2.6	2.8	3.3	3.3	4.9	5.1	7.5	11.0	11.1	11.1	
C-2010	La Salle	2	1.3	1.4	1.9	1.9	1.8	2.7	3.4	4.3	5.4	5.5	8.7	8.8	12.2	13.8	13.8
C-2022	Logan	5	1.4	1.5	2.1	2.2	2.7	3.4	4.3	4.3	6.9	7.0	10.6	10.7	12.9	12.9	14.2
C-2023	Saline	5	1.3	1.3	1.9	1.8	2.8	2.7	3.2	3.1	4.4	4.3	5.3	5.2	5.8	5.7	6.4
C-2030	Gallatin	5	1.0	1.0	1.97	1.1	1.5	1.5	1.8	3.4	3.4	2.2	2.2	2.4	2.5	2.6	3.1
C-2031	St. Clair	6	1.4	1.3	2.4	1.9	3.0	3.0	3.7	4.2	4.6	5.0	6.9	7.1	9.1	9.1	10.1
C-2034	Franklin	6	3.9	3.9	2.8	2.8	2.4	3.8	3.7	4.2	4.6	6.8	6.7	7.7	8.4	9.3	9.7
C-2037	Christian	6	1.6	1.6	1.6	1.6	2.1	2.1	3.3	3.8	3.5	4.9	6.3	7.4	9.5	10.1	11.5
C-2039	Marion	6	1.6	1.7	2.1	2.1	2.2	2.2	3.6	3.4	5.2	5.1	7.9	7.9	10.2	10.2	11.2
C-2042	Randolph	6	1.6	1.5	2.2	2.0	2.0	3.6	3.4	4.2	3.9	6.2	6.0	8.5	8.3	9.6	9.4
C-2046 ³	Knox	6	1.7	1.7	2.3	2.2	3.8	3.7	4.4	4.3	7.0	6.9	10.2	10.0	12.9	12.7	17.5
C-2059 ³	La Salle	(4)	1.4	1.4	2.0	1.9	3.0	3.0	3.5	3.4	5.2	5.1	7.8	7.7	9.8	9.7	14.1
C-2064	Gallatin	5	1.0	.99	1.4	1.4	1.5	2.1	2.1	2.1	2.3	3.0	3.0	3.5	3.4	4.2	4.2
C-2069	Saline	5	1.0	.99	1.5	1.5	2.1	2.1	2.1	2.1	2.4	3.0	3.0	3.6	3.8	4.2	4.2
C-2080	Saline	5	.93	.89	1.4	1.3	2.0	1.9	2.3	2.2	2.8	2.7	3.3	3.2	3.6	3.5	3.7

¹Weight increase, per cent.

²Residual moisture, per cent.

³Strip mine.

Local below coal No. 6.

TABLE 8.—RESIDUAL MOISTURE VALUES AFTER SECOND EQUILIBRATION (DETERMINED) AND PER CENT INCREASE IN WEIGHT OF SAMPLES DURING SECOND EQUILIBRATION. AIR BATH—NITROGEN ATMOSPHERE—NO EVACUATION

Lab. No.	County	Coal bed No.	HUMIDITY VALUES (PER CENT)							
			11.2		59.8		84.4		97.7	
			Wt. ¹	R.M. ²	Wt. ¹	R.M. ²	Wt. ¹	R.M. ²	Wt. ¹	R.M. ²
C-2009	Vermilion.....	7	1.3	1.4	3.3	3.4	7.2	7.2	12.3	12.5
C-2010	La Salle.....	2	1.2	1.4	7.7	7.8	13.1	13.4
C-2022	Logan.....	5	1.4	1.5	9.4	9.6	13.9	14.1
C-2023	Saline.....	5	1.4	1.3	5.3	5.1	6.0	6.2
C-2030	Gallatin.....	5	.49	.77	1.7	1.8	2.4	2.4	2.6	3.0
C-2031	St. Clair.....	6	1.3	1.3	6.8	6.8	9.9	9.9
C-2034	Franklin.....	6	1.8	1.7	4.8	4.7	8.5	8.4	9.8	9.9
C-2037	Christian.....	6	1.4	1.6	4.9	4.9	9.6	9.6	11.8	12.7
C-2039	Marion.....	6	1.6	1.6	5.2	5.2	9.8	10.1	12.8	12.9
C-2042	Randolph.....	6	1.6	1.5	4.2	4.1	8.2	8.3	10.6	10.5
C-2046 ³	Knox.....	6	1.6	1.7	4.2	4.3	9.3	9.5	16.5	16.5
C-2059 ³	La Salle.....	(⁴)	1.4	1.4	3.4	3.4	7.0	7.1	13.4	13.3
C-2064	Gallatin.....	5	1.0	1.1	2.3	2.3	3.3	3.3	4.1	4.1
C-2069	Saline.....	5	1.0	1.0	2.4	2.3	3.3	3.3	3.9	4.1
C-2080	Saline.....	5	.91	.90	2.1	2.1	3.0	3.0	3.8	3.7

¹Weight increase, per cent.²Residual moisture, per cent.³Strip mine.⁴Local below coal No. 6.

within the desiccators might eliminate this difficulty. Accordingly, portions of the 15 samples were equilibrated at three or four humidities in a nitrogen atmosphere, using the air-bath thermostat. The results obtained are tabulated in table 5 and shown graphically in figures 7, 8, and 9. Results obtained in this way are similar to those obtained in the former series of determinations. Table 6 presents a comparison of moisture values obtained by various procedures for these coals.

As a means of checking whether the vacuum oven procedure for determining moisture accounted, within experimental error, for the increase in weight of samples during second equilibration, a comparison was made of vacuum oven moisture values with the per cent increase in weight of the dry samples during the second equilibration. The comparison of these values for the 15 samples equilibrated in the water-bath thermostat is shown in table 7 and for those equilibrated in the nitrogen atmosphere, air-bath thermostat, in table 8. Reference to these tables will show that the vacuum oven procedure accounts, within experimental error, for all moisture taken up by the samples during equilibration.

Figures 4, 5, and 6, in which are plotted the moisture-humidity data for the 15 samples of coal equilibrated in the water-bath thermostat, show irregular curves similar to those obtained for the first six coals. These curves are difficult to extrapolate, as they are inclined very steeply upward toward the 100 per cent line in some instances and downward in others, leaving doubt as to their proper direction. The irregularities are greater than the allowable limits of deviation in the procedures used, which makes it impossible to draw smooth curves through the points. The low and intermediate humidity values have little or no influence on the slope of the curve as extrapolated, for this slope is determined by the last two high humidity values. In figures 7, 8, and 9 are plotted the moisture-humidity data for the samples equilibrated in a nitrogen atmosphere. These curves appear smoother than those of figures 4, 5, and 6 because fewer points were plotted. However, here again the slope of the curve as extrapolated to cut the 100 per cent humidity axis is determined by the last two points. It would seem, therefore, that equilibrations at low and intermediate humidities are entirely useless for extrapolation. Curves whose extrapolation

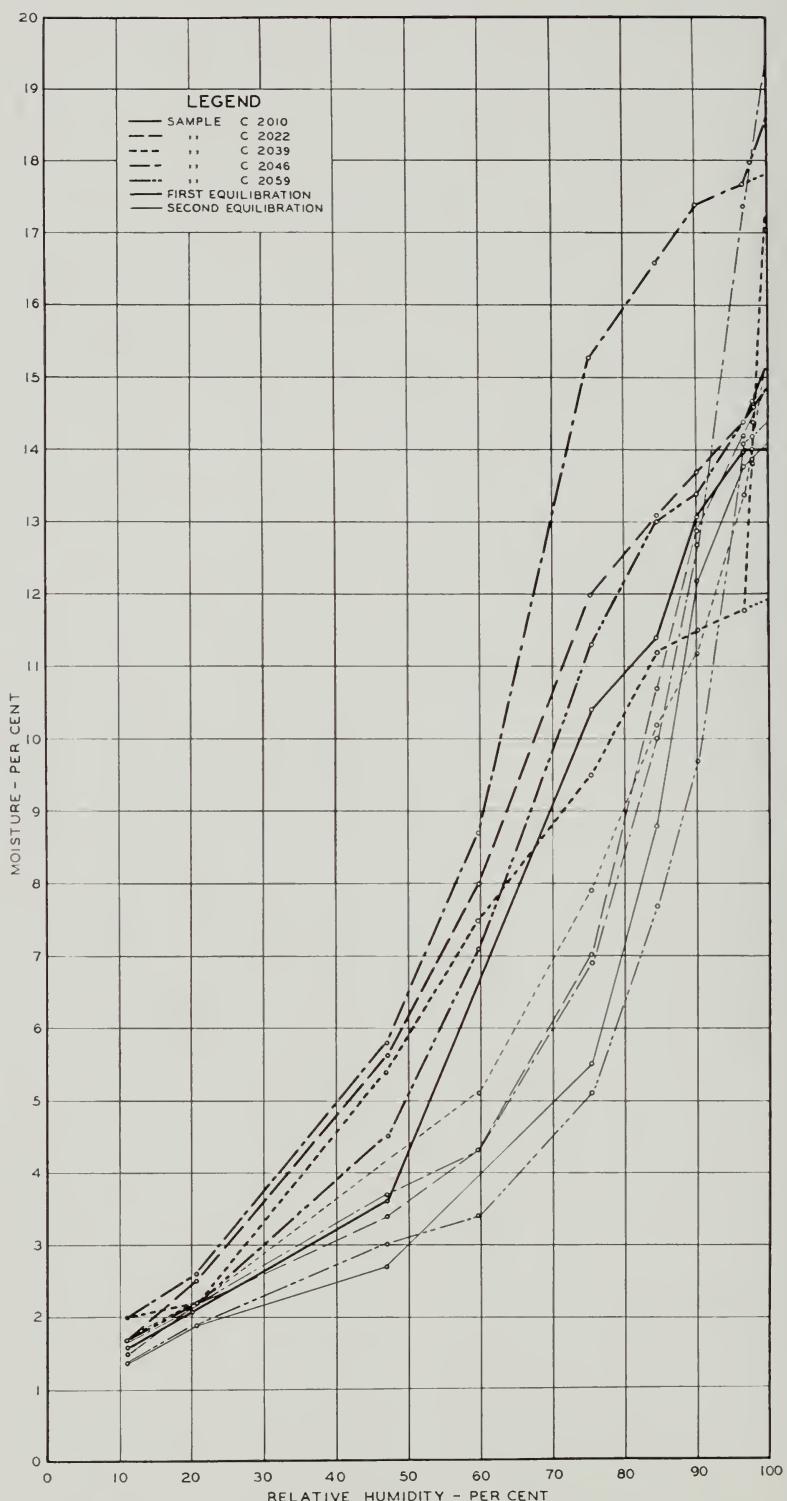


FIG. 6.—Moisture-humidity curves for high-moisture samples, desiccators evacuated, water-bath thermostat.

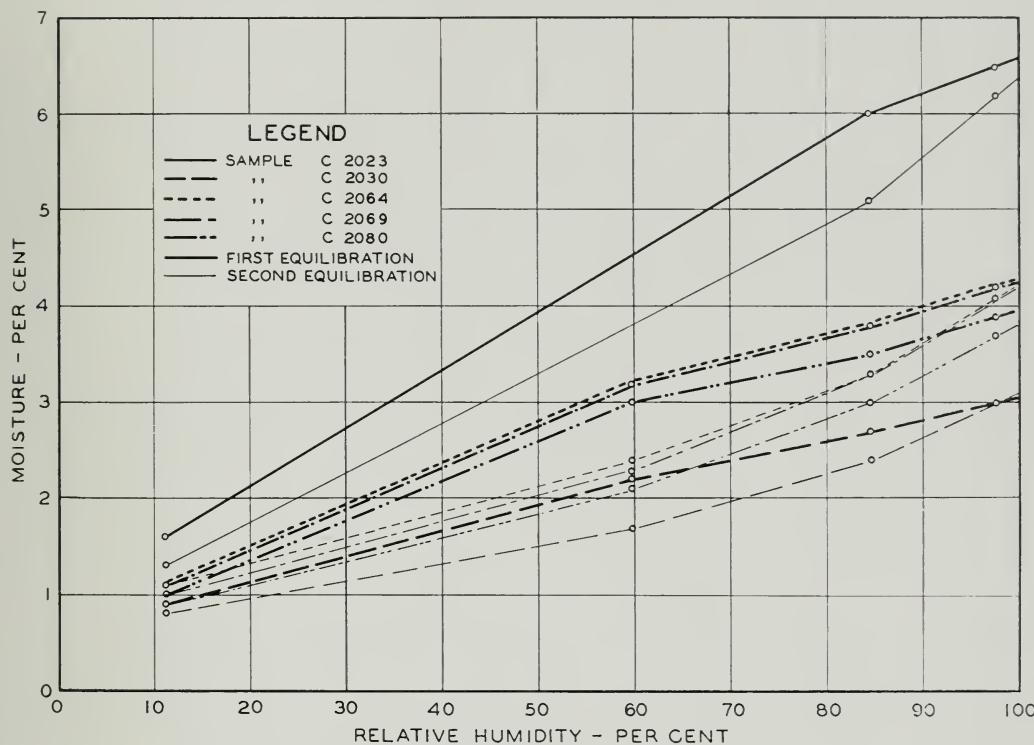


FIG. 7.—Moisture-humidity curves for low-moisture samples, nitrogen atmosphere.

slope is determined by only two or possibly three points would seem to be unreliable.

Stansfield and Gilbart (11) suggested that after characteristic curves had been run on many representative coals it was possible to calculate an average ratio between the 97 per cent humidity moisture value and the "true" moisture value obtained by extrapolation. This ratio could be used in calculating true moisture values from the 97 per cent humidity moisture values. By such a procedure equilibration at only one humidity was necessary and the value so obtained was termed moisture "by calculation." The factor as determined by Stansfield and Gilbart for this calculation was 0.986.

Similar factors have been calculated from the data in this report. The factors together with calculated moisture values are shown in tables 1, 4, and 5. Reference to tables 1 and 4 will show factors and calculated moisture values obtained from the retained moisture values at both 96.8 and 97.7 per cent humidities. The average factor of first equilibration values at

96.8 per cent humidity for the first six samples studied (table 1) is 0.971 and for second equilibration values it is 0.898. Factors for 97.7 per cent values are 0.979 and 0.930 respectively for first and second equilibration data. Considerable deviation of individual values from the averages are apparent. In calculating moisture values, average factors for first equilibration values were used for first equilibration data and average factors for second equilibration values were used for second equilibration data. The moisture values calculated from 96.8 and 97.7 per cent humidity values check reasonably well but appreciable deviations of these values from the 100 per cent extrapolated values are noted.

Factors for the 15 samples of table 4 are 0.950 and 0.952 respectively for first and second equilibration data at 96.8 per cent humidity and 0.960 and 0.957 respectively for first and second equilibration data at 97.7 per cent humidity. Again, individual factors deviate appreciably from the average factor. Calculated moisture values for the two series check very well

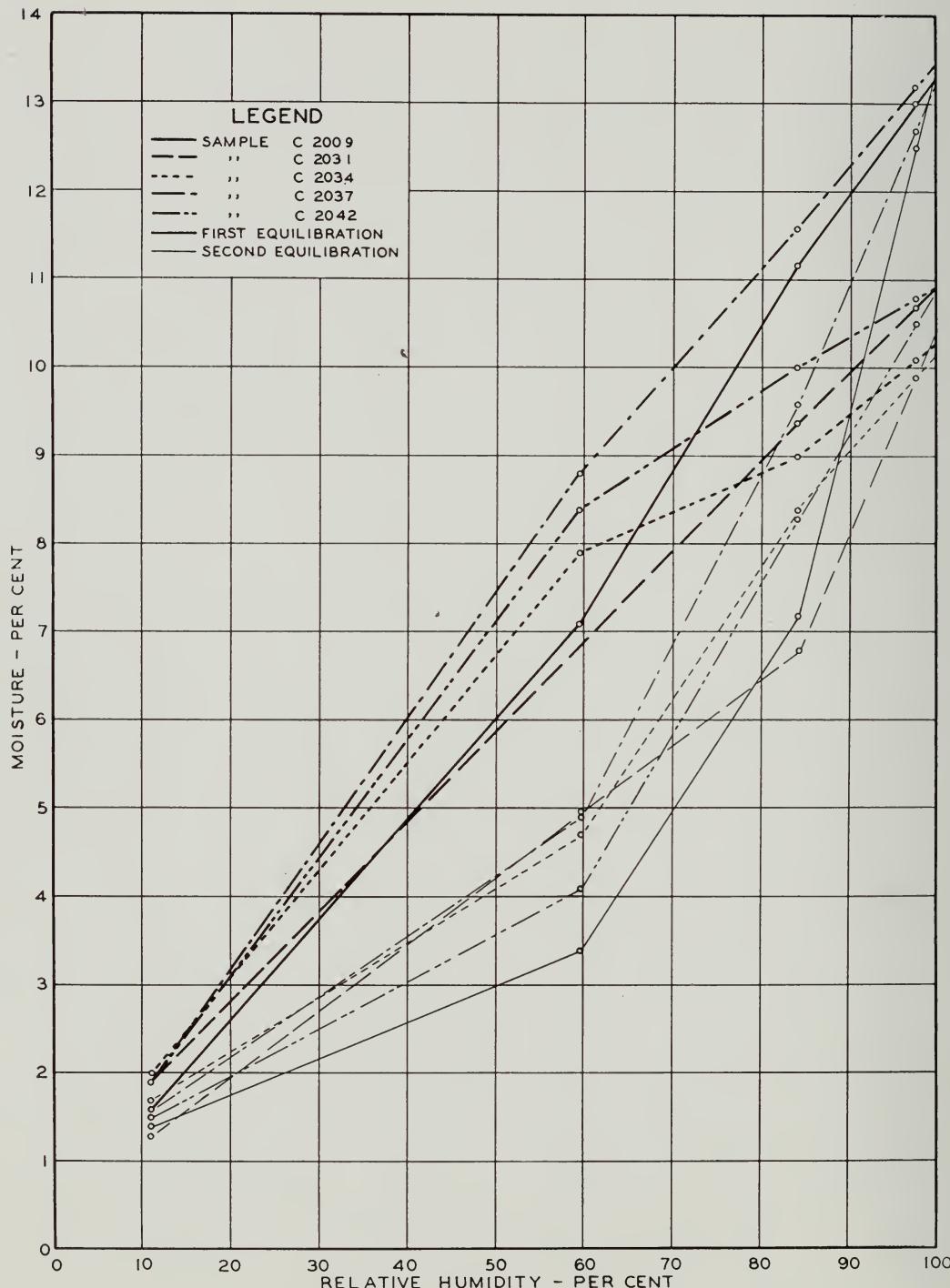


FIG. 8.—Moisture-humidity curves for medium-moisture samples, nitrogen atmosphere.

but vary considerably from the 100 per cent extrapolated values in some cases. Factors for results on samples equilibrated in a nitrogen atmosphere are available only for the 97.7 per cent humidity data. These are shown in table 5. Values of 0.981 and 0.959 respectively were obtained for first and second equilibration data. Calculated moisture values seem to check with the 100 per cent extrapolated values somewhat better than do those of tables 1 and 4.

In table 6 are tabulated moisture values for the 15 samples obtained by different procedures including first and second equilibration values obtained using evacuation and nitrogen atmosphere. In general the results obtained by various procedures seem to check reasonably well. For the most part method B, or total moisture by Air Dry loss + Regular Moisture, gave lowest results. Equilibration results for samples C-2023, C-2030, C-2064, C-2069, and C-2080 are, in general, lower than results obtained by other procedures but the equilibration results for sample C-2039 are distinctly higher than the other results for this sample.

As a test of the suitability of the moisture values obtained in this study for use in the rank classification of Illinois coals, moist mineral-matter-free B.t.u. values for all 21 coals studied were calculated on the basis of each different moisture value.

These values, together with ash, sulfur, and B.t.u. values reported on the "as received" basis according to each individual moisture value, are shown in table 9.

Reference to this table will show deviations in moist mineral-matter-free B.t.u. values in only four of the 21 coals studied, which are in the critical range for classification. Critical moist mineral-matter-free values are 11,000, 13,000 and 14,000 B.t.u. (1, 2). On sample C-1901A, methods A, B, C, and E produced moisture values leading to calculated heat values greater than 11,000 B.t.u. whereas methods D and F gave values leading to calculated heat values of less than 11,000 B.t.u. On sample C-1904A, methods A, C, and D produced moisture values leading to calculated heat values less than 11,000 B.t.u.

while methods B, E, and F gave moisture values leading to calculated heat values greater than 11,000 B.t.u. For coal C-2034, methods A, B, and C gave moisture values resulting in calculated heat values greater than 13,000 B.t.u. but methods D, E, F, G, and H gave moisture values leading to calculated heat values less than 13,000 B.t.u. On sample C-2080, all methods but one gave moisture values resulting in calculated heat values greater than 14,000 B.t.u. Method D gave a moisture value which resulted in a calculated heat value less than 14,000 B.t.u. Coals C-1901A and C-1904A are borderline coals, and agglomerating and weathering characteristics would have to be considered for classification before such deviations would be controlling factors. In three of the four cases where the heat values are around critical dividing points, the deviations of moist mineral-matter-free B.t.u. values are within experimental error and are therefore not significant.

CONCLUSIONS

The study of the equilibration procedure has led to the following conclusions:

- 1) The equilibration procedure as applied to the Illinois coals studied produces data which, when plotted, give irregular curves. Satisfactory extrapolation of these curves is impossible.
- 2) Low and medium humidity values do not appear to affect the slope of extrapolated curves.
- 3) Other procedures which are much simpler, but may involve slight modification of the standard procedure for sampling, appear to produce results suitable for classification by rank.

Although the general equilibration method does not appear suitable for determining 100 per cent humidity moisture values for rank classification, it may be useful for studying the nature of moisture in coal. The authors plan to use this method in studying the nature of the moisture of banded ingredients of representative Illinois coals in an attempt to throw further light on the nature of moisture in coal.

EQUILIBRATION METHOD OF

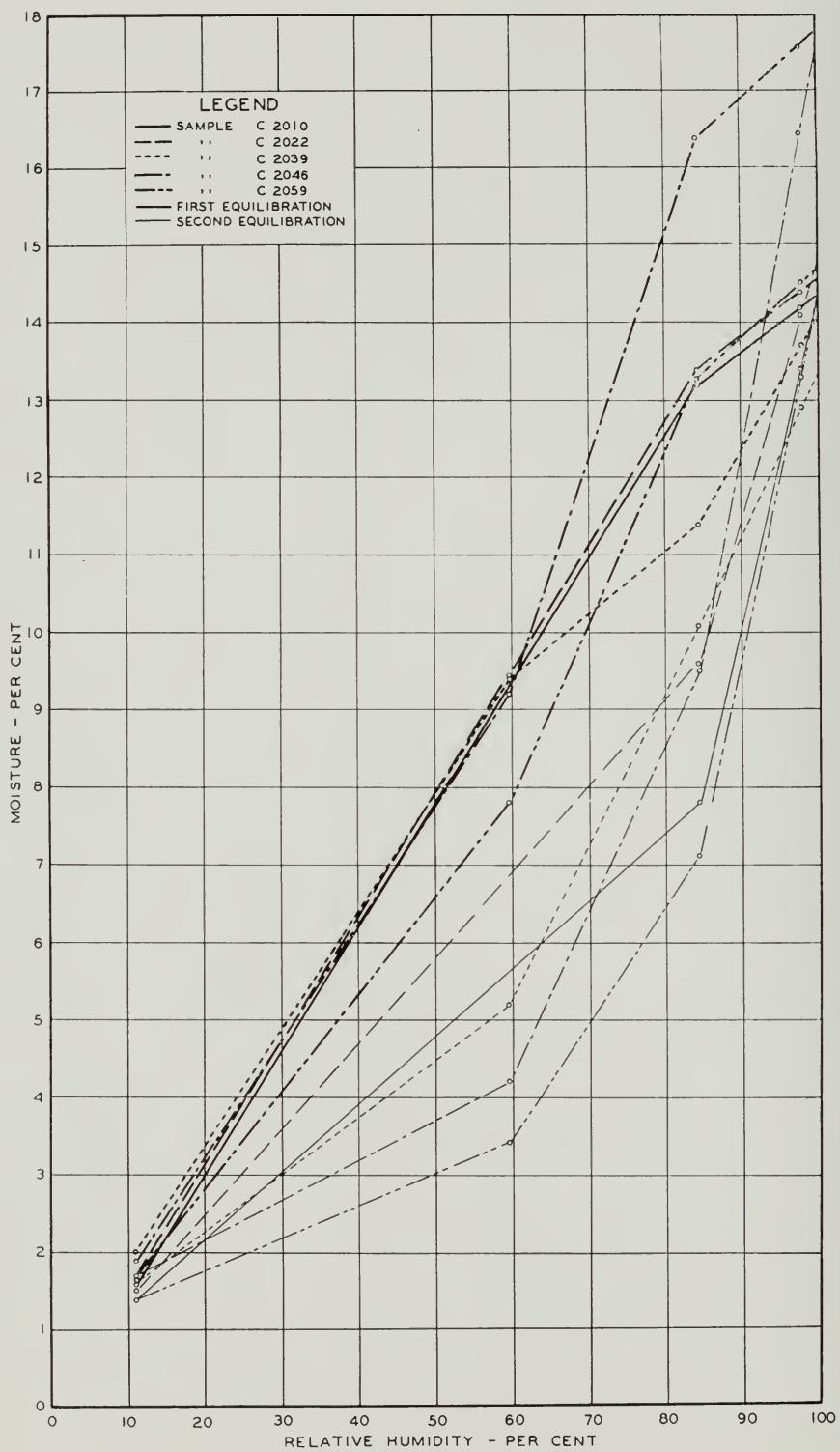


Fig. 9.—Moisture-humidity curves for high-moisture samples, nitrogen atmosphere.

TABLE 9.—MOIST MINERAL-MATTER-FREE CALORIFIC VALUES AS CALCULATED USING MOISTURE VALUES OBTAINED BY DIFFERENT METHODS. (All Coals contain less than 69 percent dry Mineral-Matter-Free Fixed Carbon)

Lab. No.	County	Coal bed No.	Method ¹	Moisture as Rec'd	Ash as Rec'd	Sulfur as Rec'd	B.t.u. as Rec'd	B.t.u. moist M.M. Free
C-1863A	La Salle.....	2	(A)	14.6	10.1	3.88	10786	12181
			(B)	14.2	10.1	3.90	10837	12240
			(C)	14.4	10.1	3.89	10811	12210
			(D)	14.7	10.1	3.87	10773	12165
			(E)	14.3	10.1	3.89	10824	12225
			(F)	14.4	10.1	3.89	10811	12210
C-1864A	Perry.....	6	(A)	10.5	10.7	3.91	11012	12535
			(B)	9.9	10.8	3.94	11086	12637
			(C)	10.2	10.8	3.92	11049	12593
			(D)	10.5	10.7	3.91	11012	12535
			(E)	10.4	10.8	3.92	11024	12564
			(F)	10.0	10.8	3.93	11074	12623
C-1900A	Saline.....	5	(A)	7.2	8.4	2.51	12333	13632
			(B)	6.6	8.4	2.52	12413	13722
			(C)	7.1	8.4	2.51	12346	13647
			(D)	7.2	8.4	2.51	12333	13632
			(E)	6.7	8.4	2.52	12400	13708
			(F)	7.0	8.4	2.51	12360	13663
C-1901A	Henry.....	6	(A)	18.4	9.7	3.84	9884	11088
			(B)	17.7	9.8	3.87	9969	11199
			(C)	18.7	9.7	3.82	9848	11046
			(D)	19.1	9.6	3.80	9779	10976
			(E)	18.1	9.7	3.85	9921	11130
			(F)	19.0	9.6	3.81	9812	10991
C-1903A	St. Clair.....	6	(A)	10.5	11.1	3.76	11098	12694
			(B)	10.6	11.1	3.75	11087	12681
			(C)	10.4	11.1	3.76	11110	12708
			(D)	10.8	11.1	3.75	11061	12651
			(E)	10.4	11.1	3.76	11110	12708
			(F)	11.9	10.9	3.70	10924	12459
C-1904A	Henry.....	6	(A)	20.7	10.0	3.25	9636	10838
			(B)	19.5	10.1	3.30	9782	11019
			(C)	20.4	10.0	3.26	9672	10879
			(D)	20.8	10.0	3.25	9624	10824
			(E)	18.6	10.3	3.34	9891	11172
			(F)	18.8	10.2	3.33	9867	11131
C-2009	Vermilion....	7	(A)	13.3	10.1	3.10	10989	12398
			(B)	12.6	10.2	3.13	11078	12516
			(C)	13.5	10.1	3.10	10964	12369
			(D)	14.0	10.1	3.08	10901	12297
			(E)	13.1	10.2	3.11	11015	12443
			(F)	13.1	10.2	3.11	11015	12443
			(G)	13.3	10.1	3.10	10989	12398
			(H)	13.3	10.1	3.10	10989	12398
C-2010	La Salle.....	2	(A)	14.3	9.0	3.03	11072	12324
			(B)	13.1	9.1	3.08	11227	12515
			(C)	14.4	9.0	3.03	11060	12310
			(D)	14.9	8.9	3.01	10995	12222
			(E)	14.0	9.0	3.04	11111	12368
			(F)	14.1	9.0	3.04	11098	12353
			(G)	14.4	9.0	3.03	11060	12310
			(H)	14.3	9.0	3.03	11072	12324

EQUILIBRATION METHOD OF

TABLE 9—(Continued)

Lab. No.	County	Coal bed No.	Method ¹	Moisture as Rec'd	Ash as Rec'd	Sulfur as Rec'd	B.t.u. as Rec'd	B.t.u. moist M.M. Free
C-2022	Logan.....	5	(A)	14.4	11.4	2.84	10566	12103
			(B)	13.8	11.5	2.86	10641	12206
			(C)	14.6	11.4	2.84	10542	12075
			(D)	15.1	11.3	2.82	10480	11988
			(E)	15.2	11.3	2.82	10468	11974
			(F)	14.9	11.3	2.83	10505	12017
			(G)	14.6	11.4	2.84	10542	12075
			(H)	14.8	11.3	2.83	10517	12031
C-2023	Saline.....	5	(A)	7.7	9.3	2.97	12103	13535
			(B)	7.7	9.3	2.97	12103	13535
			(C)	7.3	9.4	2.98	12156	13612
			(D)	8.1	9.3	2.96	12051	13476
			(E)	6.5	9.4	3.01	12261	13732
			(F)	6.3	9.5	3.02	12287	13779
			(G)	6.6	9.4	3.01	12248	13717
			(H)	6.4	9.5	3.01	12274	13763
C-2030	Gallatin.....	5	(A)	3.8	11.1	3.39	12637	14472
			(B)	3.7	11.1	3.39	12650	14487
			(C)	3.1	11.1	3.41	12729	14580
			(D)	3.7	11.1	3.39	12650	14487
			(E)	3.1	11.1	3.41	12729	14580
			(F)	3.3	11.1	3.40	12703	14549
			(G)	3.1	11.1	3.41	12729	14580
			(H)	3.1	11.1	3.41	12729	14580
C-2031	St. Clair.....	6	(A)	10.9	11.6	3.69	11008	12668
			(B)	10.4	11.6	3.71	11070	12741
			(C)					
			(D)	11.5	11.5	3.66	10934	12564
			(E)	10.4	11.6	3.71	11070	12741
			(F)	10.6	11.6	3.70	11045	12711
			(G)	10.9	11.6	3.69	11008	12668
			(H)	10.4	11.6	3.71	11070	12741
C-2034	Franklin.....	6	(A)	9.5	8.4	1.16	11799	13004
			(B)	9.4	8.4	1.16	11812	13018
			(C)	9.4	8.4	1.16	11812	13018
			(D)	10.0	8.4	1.15	11734	12931
			(E)	9.8	8.4	1.15	11760	12960
			(F)	9.7	8.4	1.16	11773	12975
			(G)	10.3	8.3	1.15	11695	12873
			(H)	10.1	8.4	1.15	11721	12917
C-2037	Christian.....	6	(A)	13.0	11.2	4.48	10618	12165
			(B)	12.0	11.4	4.53	10740	12340
			(C)	12.9	11.2	4.49	10630	12180
			(D)	13.4	11.2	4.46	10570	12109
			(E)	14.3	11.1	4.41	10460	11964
			(F)	13.4	11.2	4.46	10570	12109
			(G)	13.5	11.2	4.45	10557	12093
			(H)	13.2	11.2	4.47	10594	12137
C-2039	Marion.....	6	(A)	12.5	9.9	3.32	11001	12385
			(B)	12.3	9.9	3.32	11026	12414
			(C)	12.5	9.9	3.32	11001	12385
			(D)	13.1	9.8	3.29	10925	12283
			(E)	17.1	9.4	3.14	10422	11649
			(F)	15.2	9.6	3.21	10661	11951
			(G)	14.1	9.7	3.26	10799	12123
			(H)	13.4	9.8	3.28	10887	12239

TABLE 9—(Concluded)

Lab. No.	County	Coal bed No.	Method ¹	Moisture as Rec'd	Ash as Rec'd	Sulfur as Rec'd	B.t.u. as Rec'd	B.t.u. moist M.M. Free
C-2042	Randolph	6	(A)	10.9	10.7	2.80	11104	12616
			(B)	10.2	10.8	2.82	11191	12733
			(C)	11.1	10.7	2.79	11078	12586
			(D)	11.6	10.6	2.78	11016	12499
			(E)	10.9	10.7	2.80	11104	12616
			(F)	11.1	10.7	2.79	11078	12586
			(G)	10.9	10.7	2.80	11104	12616
			(H)	10.9	10.7	2.80	11104	12616
C-2046	Knox	6	(A)	18.0	9.1	2.95	10243	11401
			(B)	17.9	9.1	2.96	10256	11416
			(C)	17.6	9.1	2.97	10293	11458
			(D)	18.8	9.0	2.92	10144	11275
			(E)	18.6	9.0	2.93	10168	11302
			(F)	19.3	9.0	2.91	10081	11204
			(G)	17.8	9.1	2.96	10268	11429
			(H)	17.6	9.1	2.97	10293	11458
C-2059	La Salle	Local below 6	(A)	15.1	6.5	2.16	11386	12286
			(B)	15.1	6.5	2.16	11386	12286
			(C)	14.5	6.6	2.17	11466	12388
			(D)	15.1	6.5	2.16	11386	12286
			(E)	14.9	6.6	2.16	11413	12330
			(F)	14.4	6.6	2.17	11480	12404
			(G)	14.7	6.6	2.17	11440	12360
			(H)	14.3	6.6	2.18	11493	12418
C-2064	Gallatin	5	(A)	5.0	8.9	3.17	12664	14107
			(B)	5.0	8.9	3.17	12664	14107
			(C)	4.8	8.9	3.18	12690	14137
			(D)	5.1	8.9	3.17	12650	14092
			(E)	4.5	9.0	3.19	12730	14200
			(F)	4.2	9.0	3.20	12770	14245
			(G)	4.3	9.0	3.20	12757	14231
			(H)	4.2	9.0	3.20	12770	14245
C-2069	Saline	5	(A)	4.8	11.5	3.29	12229	14066
			(B)	4.8	11.5	3.29	12229	14066
			(C)	4.7	11.5	3.30	12242	14082
			(D)	5.1	11.5	3.28	12191	14021
			(E)	4.3	11.6	3.31	12294	14160
			(F)	4.7	11.5	3.30	12242	14082
			(G)	4.3	11.6	3.31	12294	14160
			(H)	4.2	11.6	3.31	12306	14174
C-2080	Saline	5	(A)	5.0	12.3	3.49	12070	14028
			(B)	5.0	12.3	3.49	12070	14028
			(C)	4.8	12.3	3.49	12096	14059
			(D)	5.1	12.2	3.48	12058	13996
			(E)	4.0	12.4	3.52	12198	14198
			(F)	4.1	12.4	3.52	12185	14183
			(G)	4.0	12.4	3.52	12198	14198
			(H)	3.8	12.4	3.53	12223	14228

^{1A} Sample ground to $\frac{1}{4}$ -inch in mine. Total moisture by Air Dry Loss + Regular Moisture.^{1B} 50-lb. mine sample crushed to $\frac{1}{4}$ -inch in laboratory. Total moisture by Air Dry Loss + Regular Moisture.^{1C} 5-gram sample crushed to 20 mesh in mine. Moisture by A.S.T.M. oven, air atmosphere, 105° C. $1\frac{1}{2}$ hours.^{1D} 5-gram sample crushed to 20 mesh at mine. Moisture by vacuum oven 3 inches Hg., N_2 atmosphere, 105° C., 3 hours.^{1E} 100 per cent humidity extrapolated moisture values—1st equilibration, evacuated.^{1F} 100 per cent humidity extrapolated moisture values—2nd equilibration, evacuated.^{1G} 100 per cent humidity extrapolated moisture values—1st equilibration, nitrogen atmosphere.^{1H} 100 per cent humidity extrapolated moisture values—2nd equilibration, nitrogen atmosphere.

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APPENDIX A

DETERMINATION OF THE VAPOR PRESSURES OF THE SATURATED
SALT SOLUTIONS USED IN EQUILIBRATION TESTS

By O. W. REES AND G. W. LAND

The problem of making vapor pressure determinations on the saturated salt solutions used in the equilibration tests was complicated by the necessity of obtaining values for them as used in the moisture study rather than in cells especially constructed for vapor pressure determinations. Following is a description of the apparatus and procedure used as well as the results obtained.

Of the many procedures available for making vapor pressure determinations two were selected as promising. The first procedure tried was the static method used by Frowein (1) which used an oil-filled siphon manometer. This was found to be unsuitable so the manometer method applied by Rayleigh (2) to the measurement of low pressure was tried. This procedure, with certain modifications, proved to be satisfactory. A description of the apparatus used in this study follows.

APPARATUS

The double-arm manometer was constructed of glass, and the two arms were connected at both the top and bottom with glass tubing. From the tube connecting the bottoms of the manometer arms, a long glass tube extended down to a mercury well which could be raised and lowered to control the mercury level in each arm. The upper connecting tube was provided with a vacuum stopcock making it possible to close off one arm from the other. Each arm was provided with a sealed-in-tungsten contact point. These points were connected through push buttons and dry cell battery to a lamp and scale galvanometer for use in levelling the manometer while enclosed in an air-bath thermostat. The manometer assembly was mounted on a framework pivoted at the center to provide tilting in either direction. Tilting

was controlled by a screw extending through the bottom of the air-bath thermostat. A mirror for projecting the angle of tilt was mounted at the hub of the tilting device half way between the two arms of the manometer. The manometer was connected to a McLeod gage by a long slightly flexible glass U-tube. This gage measured the absolute pressure in the system at the beginning of each determination. The flexible glass tube also connected to the system the desiccator containing the unknown solution. The temperature of this solution was controlled at $30^{\circ}\text{C.} \pm 0.1^{\circ}\text{C.}$ by immersing the desiccator in a water-bath thermostat. A light was mounted on a millimeter scale four meters from the manometer mirror in such a way that the angle of tilting was projected by the mirror through a glass window in the door of the air-bath thermostat on to the scale and read by means of a telescope. Figure A gives a diagrammatic sketch of the manometer assembly, side view or view along the axis of rotation of the manometer. Figure B is a diagrammatic sketch of the manometer assembly, front view or view perpendicular to the axis of rotation of the monometer. Figure C is a diagrammatic sketch of the manometer, scale, and alidade, side view. Figure D is a photographic view of the manometer assembly. This figure shows a Leeds & Northrup lamp and scale in position to read the projected tilt of the manometer. This was later replaced with a surveyor's alidade which was used for all measurements reported. A rubber tube connection to the levelling reservoir is also shown in this figure. This was later replaced with a glass tube extending straight down to the reservoir. Vacuum desiccators of the type shown in figure D were used to contain the saturated solutions whose vapor pressures were measured.

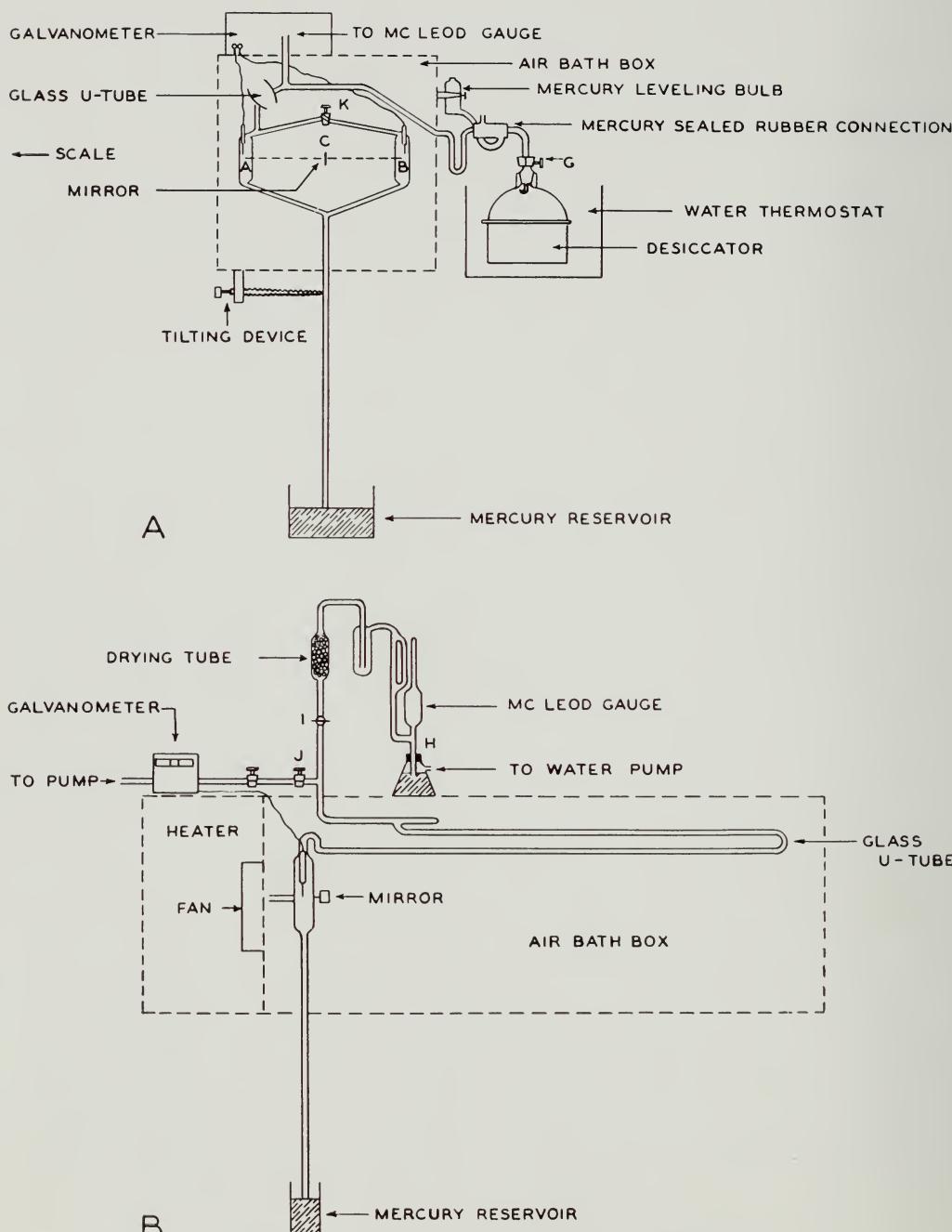


FIG. A (above).—Diagrammatic sketch of manometer assembly, side view.

FIG. B (below).—Diagrammatic sketch of manometer assembly, front view.

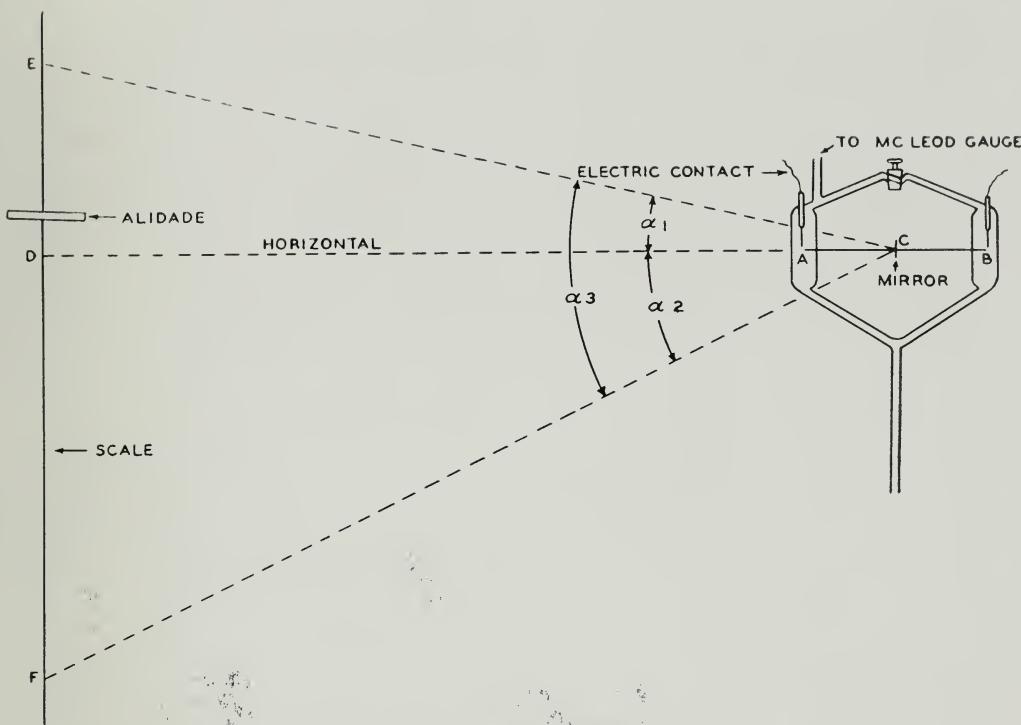


FIG. C.—Diagrammatic sketch of manometer, scale, and alidade, side view.

PROCEDURE

Saturated solutions for the vapor pressure measurements were prepared by two procedures. In the first procedure a saturated solution was prepared and placed in the bottom of the desiccator. An excess of the salt was placed in direct contact with this solution to insure complete saturation at all times. In the second procedure the dry salt was placed in the desiccator together with a small beaker of distilled water. The desiccator was closed, evacuated, and allowed to stand until the water in the beaker had passed over to the salt. While the first procedure was more rapid, it produced solutions containing more entrapped air bubbles than the second. However, it was possible to remove these air bubbles by several alternate evacuations and returns to atmospheric pressure. Duplicate determinations were made on solutions of each salt prepared by each method.

The procedure for the actual vapor pressure measurements was as follows:

The air and water-bath thermostat were brought to the desired temperature

(30°C.). The desiccator containing the saturated solution was evacuated by using an aspirator pump, and placed in the water bath. When it had reached the 30°C. temperature (determined by a small Anschutz thermometer placed inside the desiccator) it was connected to the manometer system through the mercury-sealed rubber connection (fig. A), stopcocks G and I (figs. A and B) were closed and the system was partially evacuated with a Hyvac pump. Stopcock G was then opened and the system, including the desiccator, was further evacuated. Pumping was continued for about ten minutes after which stopcock G was again closed and with stopcock I opened, the system was evacuated until the McLeod gage reading was suitably low. This value was recorded and stopcock J (fig. B) was closed. The manometer was then levelled by adjusting the tilt of the beam with the special screw (fig. A) until the contact points indicated that the mercury in both arms was equal in height in the arms. It was necessary to adjust the height of the mercury

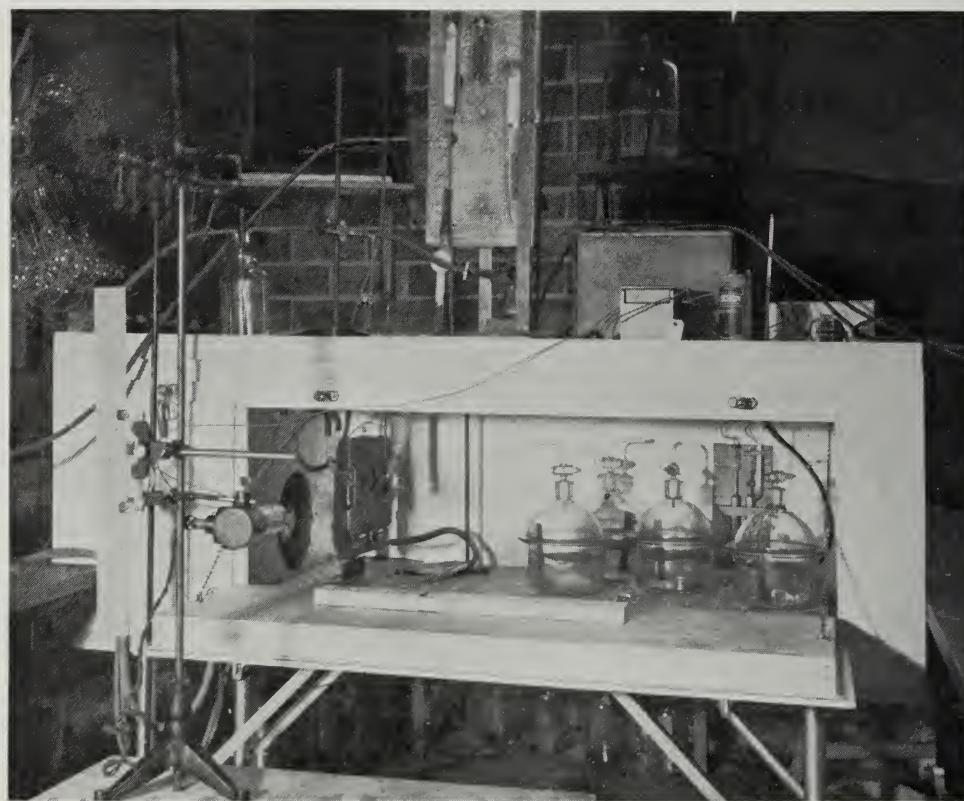


FIG. D.—Manometer assembly.

TABLE I.—EXPERIMENTAL VAPOR PRESSURE VALUES

Saturated salt solution	1 2 3 Solution prepared by direct com- bination method			4 5 6 Solution prepared by distillation method			7 Differ- ence between results from two methods	8 Average of results from two methods	9 Vapor pressures used by Stansfield and Gilbart	10 Differ- ence between columns 8 and 9				
	Vapor pressure, mm.			Vapor pressure, mm.										
	1st	2nd	avg.	1st	2nd	avg.								
KClO ₃	31.116	31.066	31.091	31.091	31.070	.021				
K ₂ SO ₄	30.801	30.751	30.776	30.751	30.785	30.768	.008	30.772	30.800	.028				
BaCl ₂ ·2H ₂ O.....	28.687	28.637	28.662	28.677	28.639	28.658	.004	28.660	28.600	.060				
KCl.....	26.796	26.890	26.843	26.879	26.874	26.877	.034	26.860	26.900	.040				
NaCl.....	23.997	24.007	24.002	23.972	23.927	23.950	.052	23.976	23.900	.076				
NH ₄ NO ₃	19.090	19.045	19.068	18.978	19.032	19.005	.063	19.037	19.000	.037				
Ca(NO ₃) ₂ ·4H ₂ O.....	14.907	14.967	14.937	14.907	14.931	14.919	.018	14.928	14.900	.028				
CH ₃ COOK.....	6.529	6.482	6.506	6.535	6.606	6.571	.065	6.539	6.365	.174				
LiCl·H ₂ O.....	3.575	3.523	3.549	3.573	3.532	3.552	.003	3.551	3.560	.009				

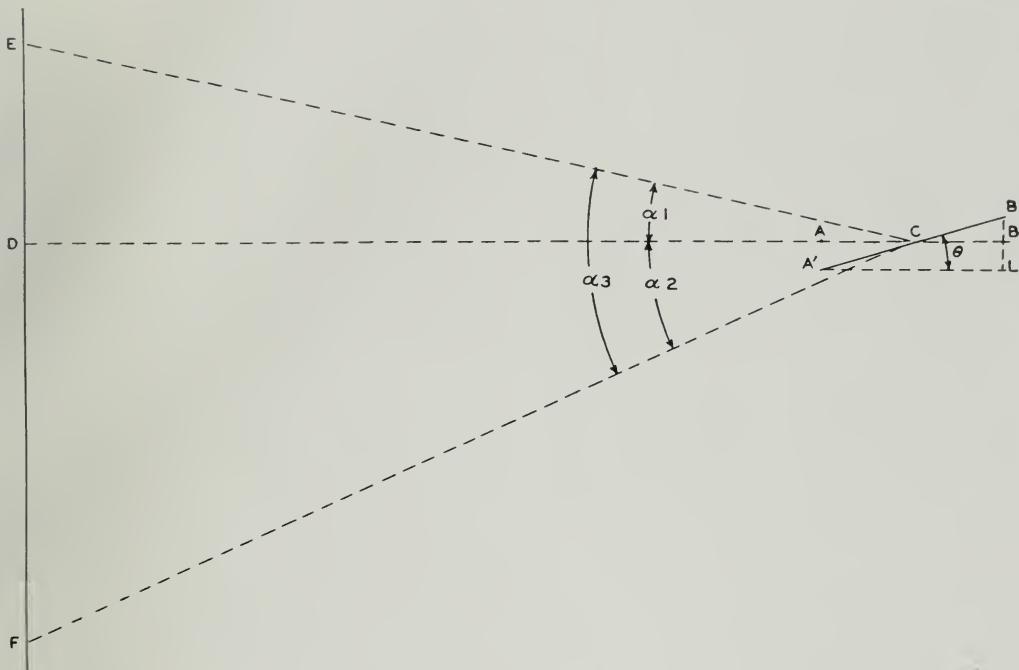


FIG. E.—Diagrammatic sketch showing the geometrical relationship involved in the calculations.

reservoir (figs. A and B) during this levelling process. When proper levelling was secured the projection from mirror C (fig. C) was read on the scale by use of the alidade. Stopcock K (fig. A) was then closed, shutting off the back arm of the manometer from the rest of the system, stopcock I was closed, and stopcock G was opened, admitting the unknown vapor pressure to the front arm of the manometer. The rocker arm was then tilted and the height of the mercury reservoir adjusted until the contact points in both arms just touched the mercury. The mirror projection was read and recorded and the manometer was adjusted at five-minute intervals until a constant reading was obtained.

From the data obtained as outlined above and from certain fixed dimensions of apparatus vapor pressure calculations were made.

CALCULATIONS

The dimensional data of the apparatus (figs. C, E) as used in this work, which were necessary for vapor pressure calculations were:

- (1) Distance AB = 290.6 mm. (by cathetometer measurement)

- (2) Distance DC = 413.39 cm.
- (3) Scale position (D) level with center of mirror = 70.10 cm.
- (4) Scale reading (E) corresponding to level position of manometer.
- (5) Scale reading (F) corresponding to tilted position of manometer (due to vapor pressure of solution).
- (6) Absolute pressure of system at beginning of measurement (McLeod gage reading).

Figure E is a diagrammatic sketch showing the geometrical relationships involved in the calculations. Distance AB is the distance between manometer arms and indicates the level position of the manometer. A'B' indicates the manometer position tilted to compensate for the vapor pressure (VP) of the unknown solution; CD is the horizontal distance from mirror C to the scale; DE and DF are distances on the scale determined respectively by the level and tilted positions of the manometer. Angle α_3 is the angle passed over by the reflected beam of light and it is equal to the sum of angles α_1 and α_2 . Values for these

two angles may be obtained by the relationships

$$\tan \alpha_1 = \frac{DE}{DC} \quad \text{and}$$

$$\tan \alpha_2 = \frac{DF}{DC}$$

Numerical values may then be obtained by reference to standard trigonometric tables. The angle θ of triangle B'A'L is the actual angle of tilt and it determines the distance B'L which measures the change in height of the mercury level in the manometer due to the vapor pressure of the saturated salt solution. By the laws of reflection, the angle passed over by a reflected beam of light is twice that passed over by the reflecting surface when the latter is rotated about an axis parallel to the surface, therefore:

$$\text{angle } \theta = \frac{\alpha_3}{2} = \frac{\alpha_1 + \alpha_2}{2}$$

Having obtained the value for angle θ and knowing the distance B'A' of triangle B'A'L it is possible to calculate distance B'L

$$\sin \theta = \frac{B'L}{B'A'} \quad \text{or}$$

$$B'L = B'A' \sin \theta$$

The vapor pressure (VP) of the solution is found by the relationship

VP = B'A' sin θ + p = B'L + p
where p is the absolute pressure of the system at the beginning of the determination.

To further illustrate this procedure a sample calculation is given:

$$AB = A'B' = 290.6 \text{ mm.}$$

$$DC = 413.39 \text{ cm.}$$

$$DE = 27.10 \text{ cm.}$$

$$DF = 60.05 \text{ cm.}$$

$$p = 0.340 \text{ mm.}$$

$$\frac{DE}{DC} = \frac{27.10}{413.39} = \tan \alpha_1 = 0.06555$$

$$\text{angle } \alpha_1 = 3^\circ 45'$$

$$\frac{DF}{DC} = \frac{60.05}{413.39} = \tan \alpha_2 = .14526$$

$$\text{angle } \alpha_2 = 8^\circ 16'$$

$$\text{angle } \alpha_3 = \alpha_1 + \alpha_2 = 3^\circ 45' + 8^\circ 16' = 12^\circ 1'.$$

$$\text{angle } \theta = \frac{\alpha_3}{2} = \frac{12^\circ 1'}{2} = 6^\circ 30''.$$

$$B'L = A'B' \sin \theta = 290.6 \sin 6^\circ 30''.$$

$$B'L = 290.6 \times .10468 = 30.420 \text{ mm.}$$

$$V.P. = 30.420 + .340 = 30.760 \text{ mm.}$$

DISCUSSION OF RESULTS

Reference to table I will show that the determined values on solutions made by the two methods (col. 1, 2, 3, 4, 5, and 6) mentioned above check very well; the average difference between the two methods being .027 mm., the largest difference .063 mm. and the smallest .003 mm. The averages of these values (col. 8) check satisfactorily those used by Stansfield and Gilbart (3). Here the average deviation is .056 mm. with the largest deviation .174 mm. and the smallest .009 mm. In only one case did the determined value vary from the reported value by more than 0.1 mm. in vapor pressure. This was in the case of the potassium acetate solution, the determined vapor pressure of which was 0.174 mm. higher than the reported value; this may have been due to some difference in the salts used.

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